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THE INTERNATIONAL NICKEL COMPANY (MOND) LIMITED

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

A History of The International Nickel Company of Canada Limited

J. F. THOMPSON and N. BEASLEY: 'For the Years to Come: A Story of International Nickel of Canada.' Published by *G. P. Putnam's Sons*, New York; *Longmans, Green & Company*, Toronto; 374 pp. Price $5.

In 1848, copper-nickel deposits were discovered in the Sudbury district of Canada; in 1902 the incorporation of The International Nickel Company under the laws of the State of New York opened the way to exploitation of the Sudbury deposits; in 1928, the International Nickel Company of Canada, Limited (incorporated in 1916 as a subsidiary company of the International Nickel Company) became the parent company; in January 1929, The Mond Nickel Company, Limited, was merged with the International Nickel Company of Canada, Limited; in Spring 1961, International Nickel commenced commercial production of nickel from the newly discovered ore deposits at Thompson, Northern Manitoba, the 75,000,000 lb. of nickel available yearly from the Thompson deposits, together with the output from the Sudbury deposits, bringing the Company's annual total production capacity to over 385,000,000 lb.

Such are the bare facts of the history of The International Nickel Company of Canada Limited, but their development by any author other than one intimately involved in the main phases of the Company's progress could have resulted in an arid book, revealing little of the personalities who contributed to the formation of the largest nickel-producing company in the world, and nothing of the insight which comes from personal recollection of the work entailed in developing the potentiality of nickel to the vital rôle which it now plays in practically every branch of industry.

Mr. N. Beasley is the author of several books on business and industry. Dr. John Fairfield Thompson, the doyen of the nickel industry, joined the Company in 1906, and has thus seen more than 50 years of progress. He was responsible for the design and operation of the first INCO Research Laboratory; became Manager of its first Technical Department, and later, as Manager of Operations, supervised the construction and initial working of the Company's Huntington Works, West Virginia, directing production of high-nickel alloys. In 1949 Dr. Thompson became President of INCO, and in 1951 his Presidency was merged with the office of Chairman. He is now Honorary Chairman of the Board.

In this book Dr. Thompson's recollection of events as they took place are used as the basis for a record of the various problems encountered, the many obstacles overcome, and the continued upward trend of the nickel industry, as represented by the most important producer of the metal.

An interesting feature is the emphasis laid on the fundamental essentials which have been primarily responsible for development of the Company's interests: (1) the existence of a potential (important nickel deposits), combined with the realization that 'an ore deposit is of no value unless someone finds a way to work it, and to sell his work, profitably and successfully', and (2) the conviction that only lack of imagination or unwillingness to work could place any physical limits on development of the industry. This attitude is reflected in the title of the book, in the manner in which the history of the Company is recorded, and in the buoyant belief that further research and hard work will make possible almost limitless extension of the usefulness of nickel.

NICKEL

A History of The International Nickel Company of Canada Limited

See abstract above.

Chemical Requirements in the Recovery of Nickel

C. A. HOCHWALT, A. E. REA and K. G. ROBB: 'Chemical Requirements—The Nickel Industry.'

Preprint of paper to Chemical Market Research Association Meeting, Pittsburgh, Nov. 1960; 13 pp. + figures and tables.

This review of the chemicals employed in the recovery of nickel is in five sections. In the first three, the authors refer to the sulphide and oxide ores in which nickel mainly occurs, draw attention to the major producers of the metal and to their production facilities, and comment on data available on free-world nickel production. Extraction and refining processes from the subject of the last of the

introductory sections. In the main section of the review, the authors discuss the chemicals used in the following nickel-processing operations: flotation operations, pyrometallurgical operations, electro-refining, chemical refining.

While the authors emphasize that the geographical distribution of mills and refineries, and the individual characteristics of a specific producer's process, preclude any accurate determination of the actual requirements in the various operations, they have, however, 'tried to determine which chemicals are used, and where, and to give a rough estimate of the quantities involved'. Flow sheets supplement the points raised in the test.

Fibre Texture of Cold-Drawn Nickel Wire

P. DAYAL and R. SHARAN: 'X-Ray-Diffraction Study of Fibre Texture of Cold-Drawn Nickel Wire.'
Eastern Metals Review, 1961, vol. 14, Feb. 6, pp. 117-8.

The texture of nickel wire, annealed and then drawn to various degrees of reduction, was studied by X-ray-diffraction techniques.

A double texture of (111) and (100) orientations resulted in the case of nickel drawn to reductions below 80 per cent. With increase in reduction, the distribution of the (100) directions decreased and that of the (111) increased, until, at a reduction of 98 per cent., the texture consisted entirely of (111), indicating that (100) is an intermediate texture unstable at higher reductions. At the surface of the wire, however, there was random orientation even at 98 per cent. reduction.

From the surface to the core of the wire the texture became increasingly oriented, and, at the core, the traces of (100) which were still present at 75 per cent. reduction disappeared, at a reduction below 87 per cent., to leave only (111). The depth from the surface at which fully stable (111) orientation was observed was found to decrease with increase in the percentage reduction.

Influence of Section Size on the Creep-Rupture Behaviour of Nickel Specimens Tested in Air

See abstract on p. 158.

Rates of Solution of Nickel and Copper-Nickel Alloys in Lead

D. A. STEVENSON and J. WULFF: 'The Solution Rate of Copper, Nickel and Their Alloys in Lead.'
Trans. Metallurgical Soc., A.I.M.E., 1961, vol. 221, Apr., pp. 279-85.

The paper records the results of work which, concerned with the effect of relative velocity at a solid/liquid interface on the dissolution kinetics of pure solid materials in liquid metals, is of interest in relation to electrode kinetics, electrolytic processes and the field of liquid-metal corrosion.

A study was made of the rates of solution of copper, nickel and three (70-30, 55-45 and 30-70) copper-nickel alloys in liquid lead, at 527° and 727°C., under dynamic conditions. The relative velocity at the solid/liquid interface was varied in the range 8·65-125 cm./sec.

Analysis of the data for the pure metals indicates that the solution rate is transport-controlled at lower velocities, but a transition to mixed control occurs at higher velocities, higher temperatures and higher percentage saturation.

The type of attack on the alloys was found to depend on the grain-size of the specimens: fine-grained material was much more prone to intergranular attack, whereas coarse-grained material (predominantly used in the investigation) was prone to intergranular attack only at the highest velocities.

Ultra-Violet Absorption Spectra of Solutions of Copper and Nickel Complexes

J. FERGUSON: 'The Ultra-Violet Absorption Spectra of Solutions of Some Copper and Nickel Complexes—Evidence for Covalent Binding.'
Spectrochimica Acta, 1961, vol. 17, Mar., pp. 316-23.

The author reports the results of an investigation of the absorption spectra of solutions of copper and nickel complexes with salicylaldehyde, salicylaldimine and N-methylsalicylaldimine.

Some of the solutions were found not to obey Beer's law, and there was strong evidence for association between the complex molecules in these instances. The spectral bands of the complexes were correlated with the degree of covalency of the metal-ligand bonds, and these were found to change when there was association or appreciable solvent perturbation. The bond changes were from nearly pure covalent to pure ionic, and it is suggested that the former are best described in terms of $4s4p^24d$-hybrid orbitals. The complexes which have ionic metal-ligand bonds did not show evidence of association, and their solutions obeyed Beer's law.

Influence of Nickel on the Chromium and Carbon Relationship in Refining Stainless Steel

See abstract on p. 161.

Determination of Nickel and Chromium in Mixtures without Prior Separation

D. H. WILKINS: ''Calcein Blue'—A New Metal-fluorechromic Indicator for Chelatometric Titrations.'
Talanta, 1960, vol. 4, May, pp. 182-4.

The colour of the complexes of EDTA with elements such as copper, cobalt, chromium, etc., has caused considerable difficulty in chelatometric titrations, but the introduction of 'Calcein' and 'Calcein W' as metalfluorechromic indicators has solved many of the problems associated with these elements. In this paper

the author gives details of a new metalfluorechromic indicator, 'Calcein Blue' (a condensation product of 4-methylumbelliferone, formaldehyde, and iminodiacetic acid), which is stated to be superior to 'Calcein W' in chelatometric titration of metal ions.

In illustration of the applications of the indicator, a procedure based on reaction rates is described for the determination of nickel and chromium in mixtures without prior separations. Reference is also made to the use of 'Calcein Blue' as an indicator for the direct titration of the alkaline-earth elements.

Preparation of Nickel for Electroplating with Nickel: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Recommended Practice for Preparation of Nickel for Electroplating with Nickel.'

A.S.T.M. Designation: B 343-60*T*.
Supplement to Book of A.S.T.M. Standards Including Tentatives, Part 2, *Non-Ferrous Metals* (*Specifications*), *Electronic Materials*, 1960, pp. 179-81.

Nickel is electrodeposited on nickel where it is desired to obtain a decorative coating of higher performance, to reclaim plated parts that are defective, or to obtain a more practicable combination of nickel electrodeposits. This recommended practice is intended to serve as a guide and not as a standard procedure. Dull nickel, semi-bright nickel, bright nickel and nickel strike are given as the types for which an overplate of nickel may be desired.

The procedures available for degreasing and electrolytic alkaline cleaning are outlined, and details are given of the following treatments employed in etching or activating the nickel surfaces: anodic treatment in concentrated sulphuric acid; anodic etching in sulphuric acid; anodic etching in a Watts-type solution; acid nickel-chloride treatment; etching by acid immersion; electropolishing treatment. Finally, attention is drawn to electroplating procedures applicable in electroplating wet freshly-plated surfaces; dry freshly-plated surfaces; polished, buffed or machine-ground surfaces; anodic-alkaline-treated surfaces; rejects with chromium coatings.

Spectrochemical Determination of Nickel in Waste Effluents from Plating Operations

See abstract on p. 147.

Application of Corrosion-Resistant Nickel Coatings by Metallizing Techniques

W. B. KEYSER: 'Metallizing Nickel Powder.'
Welding Jnl., 1961, vol. 40, Mar., pp. 229-31.

Although the suitability of nickel-plated steel for use in various industrial chemical processes is well established, the nickel coating is liable to be removed during maintenance and modification of process equipment, and the exposed metal surfaces become susceptible to chemical attack. In an attempt to overcome this problem, a programme was initiated to study the feasibility of applying a corrosion-resistant nickel coating by means of metallizing techniques (application of a metal coating to a surface by spraying the surface with molten metal). The study is reported in the present paper.

To eliminate the porosity associated with coatings produced by melting a wire in an oxy-acetylene flame and blowing the molten metal particles onto the surface in an air blast, the coatings evaluated by the author were applied using a special metal-powder spray gun. The nickel powder employed was of the following composition: nickel 99·05, silicon 0·029, molybdenum 0·02, aluminium 0·003, carbon 0·09, sulphur 0·006, boron 0·05, copper 0·09, chromium 0·05, magnesium 0·02, manganese 0·03, per cent. The average particle size was 3·85 μ.

Details are given of the work carried out to determine the most suitable technique for use in preparing the basis metal prior to metallizing and to establish the optimum metallizing procedure.

On the basis of this work, metallizing *viâ* nickel powder is deemed applicable to coating steel parts for use in service in industrial chemical processes. Like most specialized metallurgical techniques, the process has its limitations, however, and optimum results are dependent on close control of all the stages involved. These stages comprise:

(1) Degrease.
(2) Preheat to 300°–400°F. (150°–205°C.): this procedure eliminates condensation of water (from burning hydrogen and oxygen) which would otherwise cause formation of rust and reduce bond strength.
(3) Vacuum blast.
(4) Metallize.
(5) Clean with wire brush.

Metallized nickel coatings are considered to offer two advantages: (1) the equipment required is portable and renders the process applicable to reclamation of materials in isolated areas; (2) the technique enables a corrosion-resisting nickel coating to be applied over a mild-steel weld joining two nickel-plated components, and hence saves the expense involved in welding with nickel filler wire.
Its disadvantages are threefold: (1) the coating peels if it is too thick or if the surface is not properly prepared; (2) a too thin coating will not exhibit satisfactory corrosion-resistance; (3) the coating has low tensile and bond strength.

Low-Temperature Characteristics of Nickel-Cadmium Batteries

J. E. GRIFFIN: 'Investigation of Cold-Temperature Characteristics of Sonotone, 100-Ampere-Hour, Nickel-Cadmium Batteries.'
U.S. Army Engineering Research and Development Laboratories, Tech. Report 1607-*TR*, Nov. 30, 1959; 31 pp.

The test programme described was initiated to determine the suitability of nickel-cadmium batteries

for use, in place of lead acid batteries, to crank engine-generator sets at temperatures as low as −65°F. (−54°C.). In view of the satisfactory cold-temperature characteristics exhibited by nickel-cadmium batteries, and their ability to recover after discharging high currents, it was hoped that their use would obviate the need for a gasoline-fuel heater.

The tests, which were conducted on two 12-volt 100-ampere-hour batteries produced by Sonotone Corporation, and in some of which the battery was short-circuited and the internal heat generated utilized to raise the temperature of the battery electrode, included determination of: (1) discharge characteristics after cold-soaking at −65°F. (both prior to and after application of a short-circuit at the battery terminals); (2) charging characteristics at −65°F.; (3) discharge characteristics of the batteries into a low impedance.

The authors conclude that:

'(*a*) The cold-temperature discharge characteristics of a nickel-cadmium battery can be considerably improved by the application of a short circuit to provide internal battery heating.

'(*b*) The short-circuit discharge characteristics of the nickel-cadmium battery are a direct function of the state of charge of the battery.

'(*c*) The nickel-cadmium battery can be charged at a temperature of −65°F., but at a lower charging efficiency than at normal ambient temperature.

'(*d*) The nickel-cadmium battery can be utilized in winterization systems of engine generator sets, without the use of external heat, at temperatures down to −65°F.

'(*e*) Consideration should be given to the use of nickel-cadmium batteries in engine-generator-set starting systems.

'(*f*) Further tests would be required to furnish the statistical data necessary for determination of the operating and limiting characteristics of the batteries.'

ELECTRODEPOSITION AND OTHER COATING METHODS

Nickel-Coated Copper Wire for Use in Electrical Equipment: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Tentative Specifications for Nickel-Coated Soft or Annealed Copper Wire.'

A.S.T.M. Designation: B 355-60*T*.
Supplement to Book of A.S.T.M. Standards Including Tentatives, Part 2, *Non-Ferrous Metals* (*Specifications*), *Electronic Materials*, 1960, pp. 77-85.

The specifications cover three classes of 'nickel-coated, soft or annealed, round copper wire for use in electrical equipment': viz., wire with a nickel coating of at least 4 per cent. (Class 4), 10 per cent. (Class 10), and 27 per cent. (Class 27) of the total weight of the coated wire.

Requirements are stipulated with respect to the materials used; tensile properties, resistivity and dimensions of the coated wire; continuity and weight of the coating; joints and finish; inspection; methods of testing; conformance criteria; density; packaging and shipping.

In an *appendix* to the specification, details are given of the procedures to be used in the electronic and gravimetric determination of the weight of the nickel coating on the wire.

Preparation of Nickel for Electroplating with Nickel: A.S.T.M. Specification

See abstract on p. 144.

Corrosion Protection by Electrodeposited Coatings

R. WEINER: 'Corrosion Protection by Electrodeposited Coatings.'

Galvanotechnik, 1961, vol. 52, Jan. 15, pp. 2-11.

In this review of the factors involved in the corrosion protection conferred on basis metals by electrodeposited coatings (the points raised are illustrated by reference to many of the commonly electrodeposited metals, e.g., zinc., nickel/chromium, silver) the author shows how an understanding of theoretical aspects has contributed to the development of coatings of improved corrosion-resistance.

After alluding to the events which led to the recognition of the practical advantages offered by electroplating, the author discusses the conclusions drawn from the finding that when the electrodeposit and substrate are of different potential, local corrosion cells occur at pores or defects in the coating, and considers the degree to which such theoretical conclusions are applicable to corrosion behaviour under service conditions. The electrochemical and other factors involved in the corrosion-resistance of nickel/chromium coatings are then noted, and attention is directed, in particular, to the deleterious effects of the presence of porosity in the final chromium overcoating, and the present-day tendency to increase the thickness of the chromium deposit and to use crack-free coatings. The author's comments on the desirability of producing pore-free coatings are further developed in relation to the capacity of corrosion products to fill up pores under certain conditions (particularly where the service environment is relatively dry), and hence to inhibit the formation of a local corrosion cell. In subsequent sections the corrosion behaviour of cadmium coatings is discussed.

A brief survey of the practical factors governing the production of corrosion-resistant electrodeposited coatings, and an evaluation of the tests employed to assess corrosion behaviour and corrosion-resistance, conclude the review.

Double-Layer-Nickel Plating Processes

K. MÜLLER: 'Double-Layer Electroplating Processes.' *Draht*, 1961, vol. 12, Mar., pp. 103–8.

According to the author, double-layer nickel coatings owe their development to the shortage of nickel during the post-war years, when, to avoid loss of nickel during polishing, the conventional matt-nickel coatings were replaced by semi-bright nickel, which was later plated with a thin bright-nickel coating. It was found that this type of composite coating obviated the need for polishing before chromium plating, even in the case of relatively rough surfaces. Only after some time, however, was it realized that the double-layer coating confers superior corrosion-resistance compared with conventional bright-nickel deposits. This finding led to the development of plating processes developed specifically for deposition of double-layer nickel, and the first automatic plant was brought into service in the United States in 1955. The process has fulfilled expectations, and is now assuming an increasingly important rôle in the field of plating.

This review of double-layer plating processes, though relating mainly to those suitable for deposition of nickel, includes some information on double-layer chromium. (The information presented is based on the literature, and, in this connexion, attention may be drawn to abstracts of relevant papers referred to in the Electrodeposition Section of the Subject Index of vol. 33, and preceding volumes, of *The Nickel Bulletin*.) Double-layer nickel coatings comprise an initial deposit of semi-bright sulphur-free nickel and a final coating of bright nickel, the former being usually two to four times thicker than the latter, and the total thickness being similar to that of conventional bright-nickel deposits. Double-layer chromium coatings consist of an initial crack-free deposit and a final deposit of controlled crack structure of the same thickness.

The author first discusses the corrosion mechanism associated with double-layer nickel deposits, and contrasts the corrosion characteristics of double-layer-nickel+chromium coatings on iron with those of conventional bright-nickel+chromium coatings. Emphasis is laid on the factors contributing to the lateral corrosion of the bright-nickel constituent characteristic of double-layer nickel.

Comprehensive studies of the corrosion-resistance of double-layer coatings have been undertaken by various workers. Findings available to date are summarized by the author, and data are presented in illustration of the superior corrosion-resistance of double-layer nickel deposits compared with conventional single-layer coatings. The advantages offered by double-layer chromium overcoatings are briefly commented upon. Micrographs are included in the article to indicate the corrosion behaviour of double-layer and other types of coating.

In the final section of the review, the author considers the salient features of the double-layer-nickel processes developed by the following: The Harshaw Chemical Company (the 'Perflow' semi-bright + the 'Perglow' bright plating solutions); W. Kampschulte et Cie. ('DeWeKa-Doniplat' + 'DeWeKa-Superglanz' solutions); Fried. Blasberg ('N2E' + '66' solutions); Langbein-Pfanhauser Werke A.G. ('Ni-E' + 'Ni-F 61' solutions).

Suitability of Phosphoric Acid as an Etching Medium in Electroplating

M. APPERLOO, J. A. V.D. HEUVEL and H. BENNINGHOFF: 'Etching with Phosphoric Acid in the Electroplating Industry.'

Galvanotechnik, 1961, vol. 52, Jan. 15, pp. 16–20.

Data available in the literature permitting no quantitative assessment of the feasibility of using phosphoric acid as an etching medium in electroplating, the investigation reported by the authors was undertaken to determine whether subjecting the substrate to pre-plating treatment with the acid would (as suggested by some workers) adversely influence deposition or adhesion of the coating. (In this connexion, it was suggested that a phosphate film would form on the basis metal during etching in the acid, but no attempt was made by the authors specifically to determine the degree to which phosphate from the treated surface would be introduced into the solution and perhaps have deleterious effects. No such effects were, however, observed during the study.)

The hot-rolled steel plate employed as a substrate was degreased, etched for 10 minutes at 70°C. in 15 per cent. phosphoric acid containing 1 per cent. etching inhibitor (calculated on the basis of 100 per cent. H_3PO_4) and 1 g./L. of a wetting agent, and then rinsed in cold water. In complementary tests, specimens were etched in 15 per cent. hydrochloric acid for 10 minutes at 18°C. and rinsed in flowing water prior to plating. The efficacy of the phosphoric-acid treatment was assessed in relation to the adhesion of zinc, copper, matt-nickel, bright-nickel and bright-chromium coatings subsequently deposited on the etched specimens. Full details are given of the pre-plating and plating procedures employed.

The tests (carried out both on a laboratory and commercial scale) are regarded as demonstrating:

(1) Under commercial plating conditions conventional hydrochloric-acid and/or sulphuric-acid etching can be replaced by a phosphoric-acid etch.

(2) Prior treatment with phosphoric acid has no adverse effect on the adhesion of the electrodeposited coatings studied.

(3) The appearance of specimens etched in phosphoric acid and then nickel-plated is considerably better than that of those treated in hydrochloric acid.

(4) Provided that the component is rinsed in flowing water after etching, no electrically-insulating phosphate film remains on the surface of the specimens after etching in phosphoric acid. No difficulties

were encountered during electrodeposition of the metallic coatings.

The authors make the following practical recommendations:

(1) The temperature of the phosphoric acid should be selected so as to ensure that the acid does not dry on the surface of the workpiece before rinsing in water. The maximum temperature of the acid is therefore governed by the thickness of the component.

(2) The phosphoric-acid etch should, if possible, be used in combination with regenerating equipment.

Industrial Plating of Screws and Bolts

J. GARNIER: 'Industrial Plating of Screws and Bolts.' *Draht*, 1961, vol. 12, Mar., pp. 95–101.

This review of the procedures employed in the plating of screws and bolts is based on French commercial practice in electrodepositing cadmium, chromium, nickel and zinc on screws fabricated from carbon steels, alloy steels (of chromium, nickel, nickel-chromium, chromium-molybdenum, and chromium-nickel-molybdenum type) and non-ferrous metals and alloys (e.g., aluminium, brass, copper, 'Duralumin', nickel silver).

Reference is first made to French standards covering materials used for screws, and, after touching on the dimensional tolerances required after plating, the author then discusses, with particular reference to their suitability for use in plating screws, the procedures employed in the preparation of basis metals (mechanical de-scaling, pickling, electrolytic degreasing, polishing), and outlines pickling and electrolytic-degreasing solutions and conditions. General notes on the deposition of copper, brass, nickel, chromium, zinc and cadmium, and the plant employed in plating, are followed by a section in which the author gives details of typical pre-treatment and nickel- and cadmium-plating procedures used in the finishing of screws. A résumé of the factors involved in solution and deposit control concludes the review.

Spectrochemical Determination of Nickel in Waste Effluents from Plating Operations

F. L. RACINE: 'A New Method for the Rapid Analysis of Metals in Industrial Waste Effluents.' *General Motors Engineering Jnl.*, 1960, vol. 7, Oct.–Dec., pp. 11–13.

In the Truck and Coach Division of General Motors Corporation, waste effluent is analyzed, to ensure that the chemical constituents are maintained within safe limits, by chemical procedures recommended by the American Public Health Association. These procedures suffer the drawback of requiring an appreciable amount of time to complete, and it was found difficult to achieve the accuracy desired in the one part per million region of concentration. To decrease the time required for the analysis, and also to provide for greater accuracy, the Company's chemists have developed a spectrochemical method of analysis which, to date, has been employed to determine the four metals commonly used in plating operations (nickel, copper, chromium and zinc). In this article the authors give full details of the equipment and procedure employed, and present data (e.g., densitometer spectral-line data) established in the determination of the four metals.

The method is stated to be six to seven times faster than the chemical techniques previously employed. Accuracy is good in determining metals in the few parts per million normally encountered, but would, it is thought, be less satisfactory for large concentrations (e.g., in the region of 200 p.p.m.).

NON-FERROUS ALLOYS

Copper-Nickel Hardener in Ingot, Slab or Shot Form: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Tentative Specification for Copper-Nickel Alloys in Ingot, Slab, or Shot Form.'

A.S.T.M. Designation: B 358–60 *T. Supplement to Book of A.S.T.M. Standards Including Tentatives, Part* 2, *Non-Ferrous Metals* (*Specifications*), *Electronic Materials*, 1960, pp. 86–7.

The specification relates to 'copper-nickel hardener in ingot, slab or shot form'. The percentage compositional requirements prescribed for the two alloys covered (nominally 70–30 and 50–50 cupro-nickel) are given in the table below.

Element	Alloy A	Alloy B
Cu	balance	balance
Ni	28·5-31·5	49·0-52·0
Cu+Ni ..	99·0 min.	98·5 min.
Fe	0·7 max.	1·0 max.
Mn	0·25 max.	0·40 max.
Si	0·01 max.	0·01 max.
C	0·02 max.	0·02 max.
S	0·01 max.	0·01 max.
Pb	0·01 max.	0·01 max.
Sn	0·01 max.	0·01 max.
Zn	0·01 max.	0·01 max.

Other sections of the specification deal with manufacturing procedures, methods of analysis, certification, etc.

Copper and Copper-Alloy Integral-Finned Seamless Condenser and Heat-Exchanger Tubes: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Standard Specification for Copper and Copper-Alloy Seamless Condenser and Heat-Exchanger Tubes with Integral Fins.'

A.S.T.M. Designation: B 359–60.

Supplement to Book of A.S.T.M. Standards Including Tentatives, Part 2, *Non-Ferrous Metals (Specifications), Electronic Materials*, 1960, pp. 88–93.

The specification lays down requirements with respect to 'external helical, integral-finned, seamless tubes of copper and copper alloys, with unfinned-end diameters up to 1 in. inclusive, for use in surface condensers, evaporators, and heat exchangers. The fins shall be produced by the cold forming of copper or copper-alloy prime surface tube.'

The tubes may be produced in the following types of material: Admiralty metal (Types A, B, C and D); red brass; aluminium brass (Type B); aluminium bronze; 70–30 copper-nickel alloy; 80–20 copper-nickel alloy (Type A); 90–10 copper-nickel alloy; copper; arsenical copper. Details are given of the temper in which the grades must be supplied, the chemical, tensile, grain-size and dimensional requirements to which they must conform. and the expansion, flattening, mercurous-nitrate and pressure tests to which they must be subjected.

Study of the Nickel-Zinc-Carbon System

H. H. STADELMAIER and F. H. HAMMAD: 'Study of the Nickel-Zinc-Carbon System.'

Metall, 1961, vol. 15, Feb., pp. 124–5.

In a previous study of the ternary carbides of the transition metals with zinc and tin, the composition of the ternary compound occurring in the nickel-zinc-carbon system was estimated as $Ni_{64}Zn_{21}C_{15}$. In the present work the region of homogeneity of the compound has been more accurately determined.

The authors describe the nickel corner of the ternary system in relation to an isothermal section at 800°C. The composition of the cubic ternary carbide was established as $Ni_{60}Zn_{30}C_{10}$.

Lattice Spacings and Densities of Gold-Nickel Alloys

G. F. DAY: 'Lattice Spacings and Densities of Gold-Nickel Alloys.'

Jnl. Inst. Metals, 1961, vol. 89, Apr., p. 296.

In measuring lattic parameters and densities over the complete range of quenched gold-nickel alloys, Ellwood and Bagley found that measured densities at some compositions were significantly smaller than the densities calculated from the lattice parameters, a result which they attributed to lattice vacancies (finding that the number of vacancies reached maximum values at approximately 3 per cent. at 20 and 90 at. per cent. nickel). Since such large concentrations of vacancies have been found in only a few unusual alloys, the authors have measured the densities and lattice parameters of five gold-nickel alloys.

For three of them, including the 90 at. per cent. nickel alloy, a vacancy concentration of zero was found within experimental accuracy. The other two alloys contained internal porosity and results for them were discarded. The lattice parameters, unlike those determined by Ellwood and Bagley, were observed to follow a smooth curve when plotted against composition, and, on the basis of the authors' study, it appears that the vacancy concentration in gold-nickel alloys is not unusually large. The data derived from the investigation are tabulated.

Rates of Solution of Nickel and Copper-Nickel Alloys in Lead

See abstract on p. 143.

Theory of Ferromagnetism of Nickel-Copper Alloys

J. SEIDEN: 'Theory of Ferromagnetism of Nickel-Copper Alloys.'

Comptes Rendus, 1961, vol. 252, Jan. 9, pp. 249–51.

In a previous paper (see abstract in *Nickel Bulletin*, 1961, vol. 34, No. 3, p. 55) the author demonstrated that his theory of ferromagnetism of metals at low temperatures provided a satisfactory explanation of the magnetic properties of nickel. He now shows that the theory is also applicable to the low-temperature magnetic characteristics of nickel-copper alloys.

Physical Properties of Nickel-base Alloys

See abstract on p. 154.

Voltammetric Determination of Cobalt and Nickel in Hard Magnetic Alloys

R. D. DEMARS: 'Voltammetric Determination of Cobalt and Nickel in Hard Magnetic Alloys.'

Analytical Chemistry, 1961, vol. 33, Mar., pp. 3 42–4

The high cobalt concentrations encountered in hard magnetic alloys limit the number of methods suitable for analysis of this type of material. The need for a more rapid and accurate method of analyzing cobalt-nickel alloys induced the author to undertake the work described. The aims of the investigation were found to be satisfied by a procedure in which nickel and cobalt are simultaneously determined using the techniques of voltammetry with continuously varying potential. Of the several electrolytes investigated, a pyridine/potassium-chloride system gave the most satisfactory results and was utilized for all the determinations.

Current/voltage curves were obtained with an all-purpose voltammetric instrument based on analog computer amplifiers. Particulars are given of the

procedures involved in the application of the method to analysis of mixtures of cobalt and nickel in standard and unknown solutions.

Rapid Spectrophotometric Determination of Copper in Non-Ferrous Alloys

G. LINDLEY: 'The Rapid Spectrophotometric Determination of Copper in Non-Ferrous Alloys.'
Analytica Chimica Acta, 1961, vol. 24, Feb., pp. 161–6.

A survey of the literature on the determination of copper in non-ferrous alloys revealed that many of the methods employed require somewhat critical reaction conditions, often involving use of specialized reagents after initial separation of the copper or of interfering elements. It was therefore decided to develop a spectrophotometric procedure which would entail no separations and would utilize a simple reaction to produce a self-colouring copper ion.

Preliminary experiments demonstrated that cupric-chloride formation in strong hydrochloric-acid solution would produce a sufficiently sensitive reaction. In the method subsequently evolved (and described in the present paper) the absorption of the chlorocupric anion is measured spectrophotometrically at a wavelength of 950 mμ. The method is stated to be applicable to a wide variety of materials, and data are presented to illustrate the accuracy achieved in determination of copper in, *inter alia*, cupro-nickel.

Selection of Materials for Marine Applications: Economic Considerations

See abstract on p. 170.

Use of Cupro-Nickel Sheathing to Protect Power-Line Poles from Marine-Borer Attack in Marine Locations

See abstract on p. 171.

NICKEL-IRON ALLOYS

Magnetic Materials in the Electrical Industry

P. R. BARDELL: 'Magnetic Materials in the Electrical Industry.'
Published by *Macdonald & Company (Publishers), Ltd.*, London, 1960; 2nd Edition (Revised); 320 pp. Price 32/6.

The book, first published in 1955, 'is intended to be helpful to senior students in physics and electrical engineering and to physicists and engineers in industry. It seeks to bridge the gap between an academic study of the properties of magnetic materials and the limited treatment of the subject possible in most textbooks for engineers. One of its aims is to link the properties of the materials with their applications.'

The new edition brings up to date, and supplements, in a volume of manageable proportions, extensive information on both theoretical and practical aspects of the subject: in this connexion, attention may be directed to the chapter on new applications of ferrites and the extension to the chapter on magnetic recording.

The book contains a glossary of terms and units, tables showing the properties of materials, a note explaining magnetic terminology and units, and copious illustrations and diagrams. The various chapters cover: theoretical considerations; permanent-magnet materials; high-permeability (soft) magnetic materials; measurements on soft magnetic materials; power-frequency applications of soft magnetic materials; communication-frequency applications of soft magnetic materials; magnetic ferrites; magnetic recording; magnetic amplifiers; transducers; and the testing of materials by magnetic means.

CAST IRON

Ferritic S.G. Iron Castings for Valves, Flanges, etc.

AMER. SOC. TESTING MATERIALS: 'Tentative Specification for Ferritic Nodular Iron Castings for Valves, Flanges, Pipe Fittings, and Other Piping Components.'
*A.S.T.M. Designation: A*455–60*T.*
Supplement to Book of A.S.T.M. Standards, Including Tentatives, Part 1, *Ferrous Metals* (*Specifications*), 1960, pp. 414–18.

The specification covers 'nodular iron castings for valve bodies, fittings, flanges, and other parts intended for critical service and for pressure-containing parts for use at elevated temperatures, generally manufactured in advance and supplied for sale from stock by the manufacturer, jobber, or other dealer'.

The castings, which are heat-treated to produce an essentially ferritic structure containing no massive carbides and no free cementite, should conform with the following compositional requirements: total carbon 3 min., silicon 2·75 max., phosphorus 0·08 max., per cent.

Tensile requirements are:

Tensile strength	60,000 p.s.i. (min.) (26·8 t.s.i.: 42·2 kg./mm.²)
Yield strength	45,000 p.s.i. (min.) (20·0 t.s.i.: 31·6 kg./mm.²)
Elongation in 2 in.	15 per cent. (min.)

Hardness requirements are laid down, as are the tests which the castings must withstand.

Percentage Chemical Requirements for Austenitic S.G. Iron: A.S.T.M. Specification

(See abstract below)

Type	D-2*	D-2B	D-2C	D-3*	D-3A	D-4	D-5	D-5B
Total Carbon, max.	3·00	3·00	2·90	2·60	2·60	2·60	2·40	2·40
Silicon	1·50-3·00	1·50-3·00	1·00-3·00	1·00-2·80	1·00-2·80	5·00-6·00	1·00-2·80	1·00-2·80
Manganese ..	0·70-1·25	0·70-1·25	1·80-2·40	1·00 max.†	1·00 max.†	1·00 max.†	1.00 max .†	1·00 max.†
Phosphorus, max.	0·08	0·08	0·08	0·08	0·08	0·08	0·08	0·08
Nickel	18·00-22·00	18·00-22·00	21·00-24·00	28·00-32·00	28·00-32·00	28·00-32·00	34·00-36·00	34·00-36·00
Chromium ..	1·75-2·75	2·75-4·00	0·50 max.†	2·50-3·50	1·00-1·50	4·50-5·50	0·10 max.	2·00-3·00

* Additions of 0·7-1·0 per cent. of molybdenum will increase the mechanical properties above 800°F. (425°C.).
† Not intentionally added.

Mechanical Properties of Austenitic S.G. Iron: A.S.T.M. Specification

(See abstract below)

Type	D-2	D-2B	D-2C	D-3	D-3A	D-4	D-5	D-5B
Tensile strength, min.	58,000 p.s.i. (25·9 t.s.i.: 40·8 kg./mm.²)	58,000 p.s.i. (25·9 t.s.i.: 40·8 kg./mm.²)	58,000 p.s.i. (25·9 t.s.i.: 40·8 kg./mm.²)	55,000 p.s.i. (24·5 t.s.i.: 38·7 kg./mm.²)	55,000 p.s.i. (24·5 t.s.i.: 38·7 kg./mm.²)	60,000 p.s.i. (26·8 t.s.i.: 42·2 kg./mm.²)	55,000 p.s.i. (24·5 t.s.i.: 38·7 kg./mm.²)	55,000 p.s.i. (24·5 t.s.i.: 38·7 kg./mm.²)
Yield strength (0·2 per cent. offset), min.	30,000 p.s.i. (13·4 t.s.i.: 21·1 kg./mm.²)	30,000 p.s.i. (13·4 t.s.i.: 21·1 kg./mm.²)	28,000 p.s.i. (12·5 t.s.i.: 19·7 kg./mm.²)	30,000 p.s.i. (13·4 t.s.i.: 21·1 kg./mm.²)	30,000 p.s.i. (13·4 t.s.i.: 21·1 kg./mm.²)	—	30,000 p.s.i. (13·4 t.s.i.: 21·1 kg./mm.²)	30,000 p.s.i. (13·4 t.s.i.: 21·1 kg./mm.²)
Elongation in 2 in., min., per cent.	8·0	7·0	20·0	6·0	10·0	—	20·0	6·0
Brinell Hardness (3000 kg.)	139-202	148-211	121-171	139-202	131-193	202-273	131-185	139-193

Austenitic S.G. Iron Castings: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Tentative Specification for Austenitic Nodular Iron Castings.'

A.S.T.M. Designation: A 439-60*T*.

Supplement to Book of A.S.T.M. Standards, Including Tentatives, Part 1, *Ferrous Metals* (*Specifications*), 1960, pp. 409–13.

The specification relates to 'austenitic nodular castings which are used primarily for their resistance to heat, corrosion and wear, and for other special purposes'. Requirements are stipulated with respect to manufacturing conditions, chemical composition, mechanical and magnetic properties, testing procedures, etc.

The compositions prescribed for the eight grades of iron covered, and the mechanical properties laid down for each, are reproduced in the tables above.

Use of S.G. Iron in Steel Mills

C. R. ISLEIB: 'Ductile Iron in Steel Mills.'

Iron and Steel Engineer, 1961, vol. 38, Feb., pp. 83–90; disc., pp. 90–1.

In the decade since the first commercial heat was cast, S.G. iron has proved its value as a material of construction in steel-mill applications requiring strength, toughness and resistance to wear and corrosion. In 1959 over 100,000 tons of S.G. iron were used in steel mills, a consumption which is expected to triple in 1961. In this review the author outlines the advantages which S.G. iron offers in this connexion, and, in illustration, alludes to applications typical of those for which the material has been found suitable.

After a brief résumé of the metallurgical characteristics and the mechanical properties of S.G. iron, reference is made to its machinability and to the

design advantages associated with its excellent castability. In the main sections of the review the author comments on the performance of the iron in various applications in rolling mills, coke plant, sinter plant, blast furnaces, the melt shop and ingot moulds. Photographs of actual components supplement the text.

Finally attention is briefly drawn to quality control, and the factors to be taken into account in welding S.G. iron.

Pitting of Diesel Cylinder Liners

H. H. COLLINS: 'Pitting of Diesel Cylinder Liners.'
Diesel Engineers and Users Assocn., 1961, *Preprint* S279; 8 pp.+ illustrations.

The rapid pitting which occurs on the water side of some diesel cylinder liners has been the subject of much investigation since the end of World War II, but no final solution to the problem has yet been reached. Prompted by the interest which such pitting has aroused in diesel-engine manufacturers and users, the author presents in this paper a brief review of the subject, and discusses, in the light of the evidence available, the various theories advanced in explanation of the phenomenon.

Summing up the information available on the mechanism of pitting, the author concludes that a contributory factor is the production of very turbulent coolant-flow conditions adjacent to the liner surface due to high-speed vibration of the liner walls. The turbulent flow promotes the development of small sites whose potential is more anodic than the remainder of the liner surface, rather than mechanical damage to the liner (i.e., pitting is caused by a corrosion and is not an erosion process). The exact mechanism is, however, still in doubt.

Commenting on the methods available for abating pitting attack, the author refers to reports that liners made from cast irons containing small amounts of copper and chromium, or nickel, chromium and molybdenum, have been found to confer resistance to pitting under conditions which cause rapid attack on liners made from unalloyed irons. It is suggested that the small amounts of alloying elements may have modified and refined the structure of the graphite flakes and increased the modulus of elasticity, one effect of which would be to reduce the amplitude of vibration generated for an impact of given force from the piston: if this hypothesis is correct, the use of spheroidal-graphite irons with a modulus of elasticity in the region of $24\text{–}26 \times 10^6$ p.s.i. might, it is suggested, in some cases prove an effective way of reducing pitting attack. In engine tests on liners made from low-alloy and austenitic spheroidal-graphite irons, it was found that both types of S.G. iron exhibited pitting after 100 hours' running time, but to a very much smaller extent than that exhibited by an unalloyed liner, results which suggest that the spheroidal-graphite irons are less susceptible to attack than the unalloyed liner, and also that the increased corrosion-resistance of the austenitic irons and the increased stiffness of the low-alloy spheroidal-graphite irons have roughly similar effects. The observation that the high-nickel irons have a greater resistance to pitting than the unalloyed liners, even though their modulus of elasticity may be lower, indicates that the increased corrosion-resistance of the austenitic irons has an effect on pitting.

Modification of the engine design to reduce the amplitude of the vibration on the liner is deemed the course of action most likely to be successful in reducing the incidence of pitting. Apart from the use of irons of increased modulus of elasticity or of increased corrosion-resistance, nickel/chromium plating and the use of corrosion inhibitors has also been reported to have beneficial effects.

CONSTRUCTIONAL STEELS

Compilation of Short-Time Tensile and Creep Data Obtained under Conditions of Rapid Heating

See abstract on p. 156.

Influence of Carbon, Silicon and Nickel Content on the Properties of Quenched-and-Tempered High-Strength Steels

A. VON DEN STEINEN: 'Mechanical Properties of Ultra-High-Strength Structural Steels.'
DEW Technische Berichte, 1961, vol. 1, Jan., pp. 2-5.

The work reported was undertaken to determine the influence of increased silicon content, and variation in carbon and nickel contents, on the mechanical properties of high-strength low-alloy steels tempered in the range 250°–400°C.

The three groups of steel investigated comprised: (1) chromium-nickel-molybdenum steels (carbon 0·3–0·5, silicon 0·21–0·27, chromium 1·08–2·04, molybdenum 0·06–0·48, nickel 1·47–4·34, per cent.); (2) high-silicon (1·32–1·86 per cent.) chromium-molybdenum-vanadium steels with carbon contents of 0·24–0·54 per cent.; (3) high-silicon (1·62–2·01 per cent.) chromium-nickel-molybdenum-vanadium steels containing carbon 0·28–0·49, chromium 0·8–2·04, nickel 1·06–2·14, molybdenum 0·43–0·55, per cent.

The test data show that silicon contents in the range 1·3–2·0 per cent. shift the initiation of 250°–400°C. embrittlement to a tempering temperature about 100C.° higher than that associated with steels of conventional silicon content. In the case of the silicon-rich steels, therefore, use can be made of a tempering temperature of up to about 350°C. without risk of reducing the elastic limit and impact strength. At carbon contents of 0·25–0·55 per cent., a tensile strength of 160–240 kg./mm.^{2} is obtainable in the tempered condition (toughness values decrease proportionately). The tensile data derived from heat-treated specimens of small cross-section revealed no effect of nickel up to a content of 2 per

cent., but notch impact strength at low temperatures appeared to be improved by such nickel additions.

Properties of Non-Magnetizable Steels

M. KRONEIS and R. GATTRINGER: 'Non-Magnetizable Steels for Service under High Mechanical Stress.' *Stahl u. Eisen*, 1961, vol. 81, Mar. 30, pp. 431–42; disc., pp. 442–5.

A non-magnetizable steel is often subjected to service conditions which necessitate a higher performance than that usually demanded of conventional austenitic steels. A stable austenitic structure is mandatory, and, among the mechanical properties required, a high yield strength is of great importance. The yield strength of stable austenitic steels can be increased in three ways: (1) by selection of suitable compositions for use in the quenched condition, (2) by precipitation hardening, and (3) by cold working. The properties obtainable in this way are illustrated in this paper by presentation of data obtained on three groups of steel representative of those subjected to each treatment: (1) high-alloy manganese, manganese-chromium-nickel, and nitrogen-containing manganese-chromium steels, (2) high-alloy nitrogen-containing manganese-chromium-vanadium steel and a nickel-manganese-chromium-vanadium steel, (3) high-alloy manganese-nickel-chromium, manganese-chromium and chromium-nickel-manganese steels.

The major portion of the paper is concerned with the properties of cold-worked austenitic steels, and data exemplifying the influence of the temperature and degree of deformation are included in the paper. The variation in the properties and structure of the cold-worked steels after annealing at 800°C. is reported, and details are given of tests carried out to determine notch-sensitivity and fatigue characteristics.

It is shown that yield strengths of up to 60 kg./mm. can be achieved, in as-quenched steels, by increase in carbon, nitrogen, manganese and chromium contents and by additions of vanadium. Vanadium, titanium or aluminium are beneficial in precipitation-hardening steels, and, in the case of steels containing carbon 0·53, manganese 20·6, chromium 14·6, nitrogen 0·2, vanadium 1·3, per cent., heat-treatment in the range 650°–700°C. after quenching from 1160°C. proved advantageous (treatment for 6 hours resulted in yield strengths higher than 100 kg./mm.^{2}).

In the case of cold-worked non-magnetizable steels, the required austenite stability was achieved by higher alloying additions, and the authors' investigations showed that steels with higher carbon and manganese contents are strengthened to a greater degree than those with low carbon contents or with nickel additions. Optimum cold-working and stress-relieving conditions were established for steels containing: carbon 0·65, manganese 8·5, nickel 7, chromium 3·5, per cent.; carbon 0·4, manganese 18, chromium 4, per cent., plus nitrogen; carbon 0·2, chromium 12, nickel 10, manganese 6, per cent.

The fatigue tests demonstrated that the fatigue strength of smooth test pieces does not rise proportionately with increase in yield strength, and that that of notched test pieces decreases with increase in yield strength (the decrease was greater when the notch was ground than when it was turned).

Mechanical Properties of 13 per cent. Nickel Martensitic Steel at Liquid-Air Temperatures

H. SCHUMANN: 'Mechanical Properties of 13 per cent. Nickel Martensitic Steel at Liquid-Air Temperatures.' *Neue Hütte*, 1961, vol. 6, Feb., pp. 92–8.

The occurrence, in recent years, of failures due to brittle fracture at temperatures below 0°C. have been reflected in the stringent mechanical-property requirements laid down in specifications covering steels employed in pressure vessels operating at low temperatures (e.g., in liquid-gas storage). In Germany the Vereinigung der Technischen Überwachungsvereine, Essen, requires a pressure-vessel material to exhibit a D.V.M. notch impact strength of at least 3 kpm./cm.^{2} at the lowest service temperature. Up to the Second World War the required minimum value was attainable with certainty at −180°C. only in high-alloy chromium-nickel and manganese-chromium austenitic steels. In Germany cheaper manganese steels with increased carbon and decreased chromium contents were developed, but since their notch impact strength at −180°C. is only 2–3 kpm./cm.^{2}, they barely meet the permissible limits: moreover, these notch impact values are achieved only after quenching from 1050°C., a treatment which is not always possible or desirable.

In 1945 a 9 per cent. nickel low-temperature steel was developed in the U.S.A. which does not need to be quenched and exhibits good corrosion-resistance in alkaline solutions, oil-field waters and certain organic acids. The steel, after double normalizing at 900° and 790°C., followed by stress relieving* at 570°C., exhibits good strength and ductility values at −180°C. Covered by *A.S.T.M. Specification A* 353, it can be readily welded with 25-20 chromium-nickel steel electrodes, and is widely

* In this connexion attention may be drawn to papers reporting the results of tests on pressure vessels fabricated from 9 per cent. nickel steel (see abstracts and table in *Nickel Bulletin*, 1961, vol. 34, No. 3, pp. 66-7). The tests demonstrate that 9 per cent. nickel steel exhibits, in the quenched-and-tempered condition, low-temperature notch toughness equal or superior to that of the double-normalized-and-tempered material called for in present Code requirements. The Companies participating in these tests consider that acceptance of the steel in the quenched-and-tempered condition would make it less costly to produce, and hence more attractive to designers and fabricators. The steel was shown to be notch-tough with either heat-treatment and to resist brittle fracture even in the presence of the pronounced residual stresses found in as-welded vessels, a finding which should, it is felt, make it possible to omit the stress-relieving treatment now required after welding.

used for low-temperature applications in the petroleum and chemical industries. In the introductory section of the paper the author alludes to a curve indicating that the low-temperature ductility of 9 per cent. nickel steel decreases at temperatures below −180°C., whereas a 13 per cent. nickel martensitic steel not only exhibits a higher notch-impact strength than the 9 per cent. steel at −180°C., but appears to retain its ductility at even lower temperatures. Although nickel steels of this type are not produced in East Germany, it was nevertheless deemed necessary to obtain data on their characteristics, particularly since the structural changes occurring during tempering differ considerably from those encountered in the standard ferritic constructional steels. It was also considered of value to determine what advantages 13 per cent. nickel steel offers over the 9 per cent. nickel steel, and whether the double-normalizing treatment recommended for the steels is essential and economic. The study now reported was undertaken with these aims in mind.

The steel employed, prepared in a basic-lined electric-arc furnace, was of the following composition: carbon 0·06, manganese 0·81, silicon 0·28, phosphorus 0·017, sulphur 0·006, nickel 12·85, per cent. Sheet 15 mm. thick was produced for testing. Transformation points were determined, and, in initial tests, tensile and notch-impact properties of as-rolled specimens were established in the range +20° to −180°C. Subsequently the notch-impact strength of specimens in the as-rolled condition, after normalizing at 750°C., and after double-normalizing at 900° and 725°C., was determined, in the same range, as a function of tempering at temperatures from 200° to 650°C. The notch-impact strength of double-normalized specimens was studied as a function of the cooling rate after tempering at 400° and 500°C., and experiments were carried out to discover whether specimens toughened by tempering at 550°C. would be liable to embrittlement on subsequent tempering at 400°C., and whether embrittlement resulting at 400°C. would be ameliorated by tempering at 550°C.

In the final section of the paper the author presents the findings of work conducted to throw light on the processes occurring during tempering of 13 per cent. nickel steel.

The data recorded show that, in the as-rolled condition, the steel possesses good mechanical properties both at room temperature and at −180°C. Stress-relieving at 550°-580°C., followed by air-cooling under carefully controlled conditions, proved advantageous and improved low-temperature impact strength (which was raised from 6·2 kpm./cm.^{2} to 7·5 kpm./cm.^{2} at −180°C.): its principle effect, however, was to preclude embrittlement during exposure to temperatures in the region of 400°C.

Double normalizing was found to result in optimum values for low-temperature ductility, but the improvements achieved were only slight, and it is therefore considered possible to dispense with such treatment.

Compared with 9 per cent. nickel steel, the 13 per cent. material is characterized by higher low-temperature ductility and by a considerably higher yield point and tensile strength, a factor which is regarded as of significance in design. Investigations of the weldability of the steel are stated to be in progress and the results will be published in due course.

The magnetic, X-ray and metallographic studies undertaken by the author confirmed and supplemented, in the case of the 13 per cent. nickel steel, the findings of Brophy and Miller (*Trans. Amer. Soc. Metals*, 1949, vol. 41, pp. 1185-1203) that the high low-temperature ductility of 9 per cent. nickel steel after tempering at just below the conventional Ac_1 point is due to the formation of austenite rendered stable at low temperatures due to carbon and nickel enrichment.

Machining High-Strength Materials

See abstract on p. 172.

Two-Stage Isothermal Treatment of Low-Alloy Nickel-Chromium-Molybdenum-Copper Steel

G. T. HIGGINS and H. J. AXON: 'Two-Stage Isothermal Treatment of an En26 Steel.'

Jnl. Iron and Steel Inst., 1961, vol. 198, May, pp. 46-9.

Quite apart from practical correlation between isothermal and continuous-cooling modes of transformation, a comparison of isothermal transformation characteristics on direct quenching with those for an interrupted quench might be expected to provide evidence concerning changes in the chemistry of metastable austenite before actual transformation. In the experiments now described, an En26 steel was chosen for study, on the basis that with this type of material there is a distinct separation of the ferrite-pearlite and bainite reaction ranges, and also an appreciable time interval before the beginning of the ferrite reaction. The authors' main aim, to clarify the nature of the readjustments which take place in the metastable austenite prior to transformation, involved the holding of metastable austenite in the isothermal ferrite range for various times prior to transformation to bainite at a standard temperature just above M_S. Most of the work was concerned with times of holding at the high temperature which are less than the incubation period at the high temperature, but a few results are reported for experiments which involved partial transformation at the high temperature.

The steel was of the following composition: carbon 0·40, manganese 0·62, silicon 0·22, sulphur 0·13, phosphorus 0·015, nickel 2·44, chromium 0·71, molybdenum 0·61, copper 0·16, per cent.

An isothermal transformation diagram for fully homogenized specimens is presented and discussed

in relation to behaviour in the as-received, banded, condition. The kinetics of the bainite reaction at 320°C. were examined as a function of austenite pretreatment for various times and temperatures between A_3 and B_s for the fully homogenized material, and evidence is presented in support of a diffusion-controlled conditioning of the austenite in this temperature range. Clustering of carbon atoms about the dissolved carbide-forming atoms molybdenum and chromium is considered influential in this respect.

Test for Evaluating the Crack Sensitivity of Welds Produced in 'HY-80' Steel

S. I. ROBERTS: 'Evaluating Crack-Resistant Electrodes for Welding 'HY-80'.'

Welding Jnl., 1961, vol. 40, Mar., pp. 110s-12s.

The quenched-and-tempered low-alloy nickel-chromium-molybdenum steel 'HY-80' was developed as a material for use in applications (particularly in submarine construction) requiring good weldability and resistance to brittle fracture at low temperatures. The extremely high restraint entailed in welding submarine structures imposes conditions under which only electrodes of the highest performance can produce satisfactory crack-resistant welds. In this paper the author describes a test which was evolved to evaluate the crack sensitivity of electrodes, and outlines the results obtained in its application to iron-powder-coated electrodes (conforming to *A.W.S. Classification E*-9018) employed to weld 'HY-80' steel.

The comparative electrode bend test was evolved as a means of predicting the type of electrode that will perform best in production welding. Different types of weld are laid down on one plate specimen of the basis metal studied, using only one operator and ensuring that the welding variables differ as little as possible. The main purpose of the test is to install a weakness in some area of the weld or basis metal which will be revealed when the surface of the plate is subsequently over-stressed by bending.

Full details are given of the procedures used in the application of the test to assessment of the suitability of electrodes for use with 'HY-80'.

The data presented show that the test is capable of predicting the production-weld characteristics of different electrodes, and though the work and material involved precludes its use as a routine test for all electrodes, the results obtained in development work could, it is considered, justify the cost. The test may be modified for use as a criterion of weldability of plate.

Abnormally severe procedures are necessary to produce sufficient cracks for comparison of the electrodes used, and, to obtain adequate results with 'HY-80' plate, the plate must be at least 1½-in. (3·75 cm.) thick. The work described does not indicate that 'HY-80' is crack sensitive.

HEAT- AND CORROSION-RESISTING MATERIALS

Physical Properties of Nickel-base Alloys

M. E. LANGSTON and C. H. LUND: 'Physical Properties of Some Nickel-base Alloys.'

Defense Metals Information Center, Battelle Memorial Inst., Report 129, May 20, 1960; 146 pp.

This report, which summarizes data on the physical properties of 33 representative nickel-base alloys, was complied with the intention of permitting ready reference to the data available, and drawing attention to those areas in which information is lacking.

The report is in two sections. In the first, the authors tabulate the chemical compositions of the alloys covered, and outline the groups in which they have, on the basis of composition, been classified. The groups, and the alloys falling into each, are listed below.

Group I. Nickel-Copper Alloys (nickel 62–70, copper 29–30, per cent.).
'Monel' and 'K Monel'.

Group II. Nickel-Silicon Alloys (nickel 82–85, silicon 8·5–10, per cent.).
'Hastelloy D'.

Group III. Nickel-Chromium-base Alloys (nickel 56–81, chromium 10–23, iron < 9, per cent.).
'Inconel'; 'Illium R'; 'Illium G'; 'Inconel X'; 'Inconel 702'; 'Alloy 713C'; 'Nimonic 75'; 'Nimonic 80'; 'Nimonic 80A'; 'DCM Alloy'.

Group IV. Nickel-Chromium-Iron Alloys (nickel 43–69, chromium 14–27, iron 8–27, per cent.).
'Hastelloy F'; 'Hastelloy X'; 'Hastelloy R-235'; 'NA-22-H'; 'Inconel 901', 'D-979'.

Group V. Nickel-Chromium-Cobalt Alloys (nickel 40–62, chromium 10–21, cobalt 7·5–34, per cent.).
'Waspaloy'; 'Nimonic 90'; 'Nimonic 95'; 'Nimonic 100'; 'Inconel 700'; 'Udimet 500'; 'Udimet 700'; 'Unitemp 1753'; 'M-252'; 'René 41'; 'Nicrotung'.

Group VI. Nickel-Molybdenum Alloys (nickel 51–72, molybdenum 15–30, per cent.).
'Hastelloy B'; 'Hastelloy N'; 'Hastelloy C'.

In the second and main section of the report the authors define the terms used to describe the physical properties covered, outline the status of the available data, and present data sheets and graphs for each of the alloys. The data relate to the following properties: melting range, density, specific heat, thermal conductivity, thermal diffusivity, thermal expansion, emissivity, modulus of elasticity, Poisson's ratio, magnetic permeability, electrical resistivity. A typical data sheet includes brief background notes on the alloy, information on physical properties, references to producers, and a list of sources from which the data were obtained.

It is emphasized that the report is not intended for use as a design manual: most of the data are

A Summary of the Characteristics of Air-Melted and Vacuum-Melted Alloys

(See abstract below)

Melting Variables				Product Characteristics				
Melting Method	Temperature	Pressure	Time Metal Molten	Hot Workability	Gas Content, p.p.m.	Cleanliness	Segregation	Properties
Air	2500°-3100°F. (1370°-1705°C.)	1 atm.	2-3 hours	Poor to fair	H 4-20 O 10-150 N 30-500	Fair	Normal	Normal
Vacuum-induction	2500°-3100°F. (1370°-1705°C.)	1 to 100 microns	1-5 hours	Good	H <1 O 4-10 N 3-50	Very clean	Normal	Improved by 20 to 200 per cent.
Consumable-electrode vacuum-arc remelting								
From air-melted electrode	2500°-8500°F. (1370°-4705°C.)	5 to 500 microns	15-30 minutes	Good	H 1-2 O 6-30 N 30-100	Clean	Relatively free	Improved by 10 to 100 per cent.
From vacuum-induction-melted electrode	2500°-8500°F. (1370°-4705°C.)	5 to 500 microns	15-30 minutes	Very good	H <1 O 1-5 N 2-20	Extremely clean	Relatively free	Improved by 30 to 300 per cent.

typical and some are preliminary, and, while certain of the physical properties may be regarded as inherent in the alloy composition, others vary considerably with the condition in which the alloy is supplied and evaluated.

Design Data on Nickel-base Alloys for Aircraft and Missiles

R. J. FAVOR, D. A. ROBERTS and W. P. ACHBACH: 'Design Information on Nickel-base Alloys for Aircraft and Missiles.'

Defense Metals Information Center, Battelle Memorial Inst., Report 132, July 20, 1960; 152 pp.

The design data assembled in this report cover mechanical and physical properties of significance in relation to the U.S. design document *MIL-HDBK-5* ('Strength of Metal Aircraft Elements'), and are presented in a format consistent with the intention to submit them, where appropriate, for inclusion in the document.

Four groups of nickel-base alloy are represented. The specific alloys falling within each group are listed below.

(1) *Matrix-Strengthened-type Alloys*
'Inconel'; 'Hastelloy B'; 'Hastelloy C'; 'Hastelloy W'; 'Hastelloy X'.

(2) *Precipitation-Hardenable-type Alloys (Air-Melted)*
'Inconel W'; 'Inconel X'; 'Inconel 700'; 'Inconel 702'; 'Inconel 901'.

(3) *Precipitation-Hardenable-type Alloys (Vacuum-Melted)*
'Hastelloy R-235'; 'M-252'; 'René 41'; 'Udimet 500'; 'Udimet 700'; 'Waspaloy'.

(4) *Casting Alloys*
'Inconel 713C'; 'GMR-253'.

The data are introduced by a section in which the authors briefly comment on general metallurgical considerations and factors involved in forging, cold forming, machining, welding and brazing the alloys. Data tabulated on the tensile, compressive, shear and bearing properties of a specific alloy are preceded by notes on relevant background information (e.g., material specifications) and followed by design curves showing the effect of temperature on these properties.

At the time of publication no guaranteed minimum or specifications properties were available for many of the alloys covered: these were, however, included in the report as potential materials for airframe applications. The sources of the data presented have been intentionally omitted.

Mechanical Properties of Air-Melted and Vacuum-Melted Steels and High-Temperature Alloys

D. C. LUDWIGSON and F. R. MORRAL: 'A Summary of Comparative Properties of Air-Melted and Vacuum-Melted Steels and Superalloys.'

Defense Metals Information Center, Battelle Memorial Inst., Report 128, Mar. 28, 1960; 114 pp.

The large-scale commercial application of vacuum-melting processes was rendered possible only by the development of efficient pumping systems during and after World War II. The object of the report is to present, under a single cover, all the information permitting comparison of the properties of air-melted and vacuum-melted steels and high-temperature alloys available to the Defense Metals Information Center on January 1, 1960. No attempt has been made to compare different alloys and the data are confined to mechanical properties: information on inclusion content, gas analysis, etc., is included only in conjunction with mechanical-property data.

The three sections of the report are concerned, respectively, with vacuum pouring, vacuum-induction melting, and consumable-electrode vacuum-arc remelting. The materials for which data are given are listed below, in the classifications in which they are considered.

Low-Alloy Steels
Nickel-molybdenum-vanadium steel; nickel-chromium-molybdenum-vanadium steel; chromium-molybdenum-vanadium steel; A.I.S.I. 4340; A.I.S.I. 52100; A.I.S.I. 6150; A.I.S.I. 9310; A.M.S. 6434; 'Tricent'; 'Air Steel' ('X200'); 'UHS 260'; 'Hy-Tuf'; A.M.S. 6263; A.M.S. 6470 ('Nitralloy'); A.M.S. 8620.

Chromium Hot-Work Die Steels
Chromium-molybdenum-vanadium steel to H-11 specification; 'Crucible 56'; 'Vascojet 1000'; 'Unimach II'; Potomac A'; 'Dynaflex'.

Stainless Steels
A.I.S.I. 403, 410, 422, 430 and 446 chromium steels; 'AM-355'.

Iron-base Heat-Resisting Alloys
'A-286'; 'N-155'; W-545'.

Nickel-base High-Temperature Alloys
'Waspaloy'; 'M-252'; 'Hastelloy R-235'; 'GMR-235'; 'GMR-235D'; 'Nimonic 100'; 'Guy Alloy'; 'Inco 700'; 'C-242'.

Cobalt-base High-Temperature Alloys
'G-34'; 'HS-31'; 'HS-151'; 'HE-1049'.

The data compared relate to the following properties (though data on all these properties are not necessarily included for all the materials covered): tensile, fatigue, hardness, impact (including ductile-to-brittle transition temperatures), stress-rupture. Since the value of the comparative data recorded is directly related to corresponding information on specimen orientation, heat-treatment, etc., data on the following were included where available: size of heat; number of heats tested; size of ingot; number of ingots tested; forging reduction; specimen orientation; specimen position; commercial form of material from which specimens were taken; heat-treatment and/or strength level; chemical analysis; gas analysis; inclusion rating; testing conditions.

The data included in the report are deemed to warrant the following conclusions:

'(1) The reduction in hydrogen content of large heats of low-alloy steels effected through vacuum pouring can lead to improved ductility.

'(2) The reduction in gas, inclusion, and impurity content of low-alloy or chromium hot-work die steels that can result from either vacuum-induction melting or consumable-electrode vacuum-arc remelting can lead to improved fatigue and impact properties, higher ductility, and better behaviour under multiaxial stresses, but seldom improves tensile strength.

'(3) The transition temperature, rupture strength, and elongation of ferritic and martensitic stainless steels may be improved by vacuum-induction melting.

'(4) The improved cleanliness, the better control of composition, and the modification of composition enabled by vacuum-induction melting have led to improved stress-rupture, fatigue and tensile properties in nickel-base superalloys.

'(5) The tensile and rupture strengths of cobalt-base superalloys are affected but little by vacuum-induction melting, although rupture ductility is improved significantly.

'(6) The improved homogeneity of segregation-sensitive hardenable stainless steels and iron-base superalloys that results from consumable-electrode vacuum-arc remelting leads to improved tensile strength, ductility, fatigue strength, and stress-rupture properties in these alloys.

'Several examples are available to show that improved properties do not accrue automatically through vacuum melting, but only through careful vacuum-melting practice.

'Table 1 (reproduced on p. 155) is a generalized summary of the property improvements that can be effected through vacuum pouring and vacuum melting of ferrous alloys.'

Compilation of Short-Time Tensile and Creep Data Obtained under Conditions of Rapid Heating

D. P. MOON and W. F. SIMMONS: 'Selected Short-Time Tensile and Creep Data obtained under Conditions of Rapid Heating.'
Defense Metals Information Center, Battelle Memorial Inst., Report 130, June 17, 1960; 88 pp.

The development of supersonic aircraft and missiles exposed to aerodynamic heating during flight has focused attention on the need for design data on the influence of short-time exposure at high temperatures on the properties of sheet materials employed in aeronautical structural applications. In this report the authors have summarized the data available in a series of 64 curves.

Stress/temperature curves were found to be the most practical method of presenting the data, and, for the sake of uniformity, an attempt was made to record tensile-strength and yield-strength data at strain rates of 0·00005, 0·01 and 1 inch per inch per second, and creep and rupture data as the stress to produce 1 per cent. creep strain or rupture in 1 second, 10 seconds, 1 minute and 10 minutes. The data incorporated were derived from tables and curves of stress *versus* temperature or time. Data obtained directly from tables were averaged, and are shown as points on the stress/temperature curves. In a few instances, tabulated data were plotted and selected data obtained from these curves.

Since rapid-heating tests have been employed primarily in the evaluation of sheet materials, comparative data for materials in bar form are not included in the report, and since the self-resistance heating method usually employed in this type of test produces questionable values of ductility

(due to localized overheating immediately prior to rupture), ductility data were disregarded.

The materials and data were selected for inclusion on the basis of general interest in the information and its suitability for graphical presentation. Reference to data omitted is, however, made in a *bibliography* which, comprising 121 items, covers all the sources of information revealed during preparation of the memorandum. Data are presented on 28 materials: three aluminium alloys; six titanium alloys; two alloy steels (A.I.S.I. 4130 and 'USS Cor-Ten'); three hot-work tool steels 'Peerless 56', 'Thermold J' and 'Chro-Mow'); six chromium-nickel stainless steels (A.I.S.I. Types 301, 304, 316 and 321, 'AM-350', and '17-7 P.H.'); and eight high-temperature alloys (the chromium-nickel-iron-base alloy 'A-286' and the chromium-nickel-cobalt-iron-base alloy 'N-155'; the nickel-base alloys 'Inconel X', 'Waspaloy', 'Hastelloy W', 'Udimet 500', 'Nimonic 90'; the cobalt-base alloy 'Haynes 25').

Phase Equilibria and Elevated-Temperature Properties of Alloys in the Ni_3Cr-Ni_3Al System

C. M. HAMMOND, R. A. FLINN and L. THOMASSEN: 'Phase Equilibria and Elevated-Temperature Properties of Some Alloys in the System Ni_3Cr-Ni_3Al.'

Trans. Metallurgical Soc., A.I.M.E., 1961, vol. 221, Apr., pp. 400–5.

Although a number of investigators have demonstrated the existence of γ' in high-temperature alloys based on the nickel-chromium-aluminium system, most of the specimens studied contained large quantities of other elements which gave rise to appreciable amounts of complicating phases such as carbides. The precise rôle of γ' has been rendered more problematical by the suspected existence of an ordering action in the γ matrix itself, γ_o. It appeared from previous studies that aluminium in nickel-chromium alloys would raise the ordering temperature of the matrix from 540°C. at Ni_3Cr to 1300°C. at Ni_3Al, an assumption which is supported by the finding that the matrix phase in the parallel nickel-iron-aluminium system is ordered to very high temperatures. Their survey of the literature prompted the authors to conduct an investigation of the simple nickel-chromium-aluminium system, using alloys of the highest available purity as a means of avoiding the complication of foreign phases and isolating the structures present in the system. The results of the study are presented in this paper.

The chromium and aluminium contents of the alloys prepared for examination ranged from 8·04 to 19·49 and 1·64 to 9·93 wt. per cent., respectively. Procedures were developed to study γ-γ_o and γ-γ' equilibria, and specimens were subjected to extensive metallographic studies by electron microscopy. X-ray-diffraction diagrams were obtained at both elevated and ambient temperatures. In a complementary phase of the work, the authors carried out creep and tensile tests on alloys of the nickel-chromium-aluminium system.

The data presented show that, within the compositional limits investigated, no long-range ordering of the γ phase occurred in the temperature range 893°–1073°C. The high rate of formation of the ordered γ' phase upon quenching was confirmed (this phenomenon is regarded as having previously retarded the understanding of the phase diagram). Specimens with controlled amounts of γ' exhibited up to a six-fold improvement in yield strength and creep strength at 750°C. compared with single-phase nickel-chromium-aluminium alloys.

Metallurgical Characteristics of 'A-286'

H. BROWN: 'Metallurgical Characteristics of 'A-286' Alloy.'

Defense Metals Information Center, Battelle Memorial Inst., Memorandum 59, July 26, 1960; 7 pp.

Fabrication of the complex chromium-nickel precipitation-hardenable heat-resisting steel 'A-286' can involve many difficulties unless the metallurgical characteristics of the material are fully taken into account. It has, for example, been noted that, in the absence of boron, a soft weak lamellar-type precipitate can result after age hardening, an effect which is accentuated if cold working precedes ageing (see abstracts of relevant papers in *Nickel Bulletin*, 1958, vol. 31, No. 9–10, pp. 269–70). In the presence of this precipitate, bulging has occurred during pressure testing of assemblies, stress-rupture values are very low, and creep rate increases greatly.

Intensive investigation of many heats of material has resulted in conflicting reports: some of the workers attempting phase identification concluded that the precipitate was definitely Ni_3Ti; some could find no nickel in the precipitate. The results of other work, indicating that two types of precipitate are involved, would, however, appear to resolve the discrepancy between the two findings. In the first part of this memorandum the author discusses the occurrence of the two precipitates, and outlines the evidence from which he tentatively concludes that the lamellar type (formation of which can be suppressed by a minimum addition of 0·001 per cent. boron) is Ni_3Ti, and that the other precipitate, of 'herringbone' type (which forms in strained areas after ageing), is probably some form of M_6C. He then goes on to consider the behaviour of 'A-286' during ageing, briefly discusses the effect of a too high boron content on weldability and forgeability, and refers to a welding test suitable for evaluating hot shortness.

Structural Study of a Nickel-base Alloy during Recrystallization after Cold Working

C. APERT: 'Influence of Cold Working on the Structure of an Age-Hardenable Heat-Resisting Alloy after Heat-Treatment at Various Temperatures.'

Comptes Rendus, 1961, vol. 252, Feb. 20, pp. 1152–4.

The metallographic study reported was undertaken to determine the effects, on the structural characteristics of a typical nickel-base age-hardenable alloy

(composition: chromium 20, cobalt 20, titanium 2·7, aluminium 1·5, per cent., remainder nickel), of recrystallization heat-treatment at various temperatures after cold working.

The alloy was solution-treated at 1080°C., aged at 750°C. (to precipitate γ' phase), subjected to various degrees of cold work, and then exposed to long-time heating at pre-determined temperatures.

On the basis of the subsequent metallographic examination, it is concluded that a fine degree of recrystallization occurred, even at temperatures in the region of 700°C. and at times of 100–200 hours. Within the recrystallized grains, or sometimes at the grain boundaries, relatively voluminous globules were in all cases found to be present in a matrix free from precipitates. In a micrograph of a region in course of recrystallization at 900°C. (which is presented in illustration of this effect) the unrecrystallized regions are seen to contain a relatively coarse γ' precipitate, while the recrystallized grains contain rounded particles in a matrix free from visible precipitates.

The author advances a tentative explanation of the phenomena observed.

Influence of γ' Particle Size on the High-Temperature Properties of Nickel-base Age-Hardenable Alloys

D. L. SPONSELLER and R. A. FLINN: 'The Effect of Gamma Prime Particle Size upon the High-Temperature Properties of Nickel-base Alloys.'

University of Michigan, Industry Program of the College of Engineering, Report IP-467, Sept. 1960; 43 pp.

Although control of the amount and size of precipitation-hardening particles has been used to advantage in virtually every heat-resisting-alloy system, the information available on the dependence of high-temperature strength and creep rate on the characteristics of the precipitated dispersion is relatively scarce. The investigation reported by the authors was therefore undertaken in an attempt to throw light on the influence of γ' characteristics (in particular, the particle size and the relative amount of the γ' dispersion) on the high-temperature strength of nickel-base alloys. In complementary experiments, coherency effects were examined as a function of various constituent elements.

The nickel-chromium-aluminium system (at 75 at. per cent. nickel) upon which the study was based was selected as permitting readier control of dispersion characteristics. Phase relationships in this system were examined by TAYLOR and FLOYD (*Jnl. Inst. Metals*, 1952-53, vol. 81, May, pp. 451–64: see abstract in *Nickel Bulletin*, 1953, vol. 26, No. 5, p. 80) and the reference temperature of 750°C. used by these investigators was also employed throughout the present research.

Thirteen heats of alloy were prepared: four air-melted heats were employed for coherency studies; the remainder were vacuum-melted and used to investigate γ' dispersion characteristics. Chromium and aluminium contents were varied in the range 4·79–17·9 and 3·77–14·3 at. per cent., respectively. Two of the heats contained alloying additions of 2·2 and 4·47 at. per cent. titanium, and another two contained 5·0 and 9·3 at. per cent. manganese.

Specimens were solution-annealed by one of three techniques, and then aged for 24 hours at 750°C. (followed, in one case, by 24 hours at 650°C.). Hardness measurements were carried out, and specimens were subjected to examination by electron-microscopical and X-ray techniques. Creep-rupture and short-time-tensile tests were conducted at 750°C.

The results of the study are presented in sections concerned with: (1) the characteristics of γ' precipitation (effect of composition; influence of precipitating conditions on the characteristics of the γ' precipitate); (2) effect of dispersion characteristics on high-temperature properties (effects of the relative amount of γ'; influence of particle size on creep properties); (3) coherency-stress studies (phase equilibria; lattice-parameter measurements). Consideration of these data leads the author to the following principal conclusions:

Cooling rates from above the solution temperature required to develop maximum room-temperature hardness increase with increase in aluminium content.

Room-temperature hardness, as well as tensile strength and creep strength at elevated temperatures, increases initially as the aluminium content is raised.

An exhaustion effect is noted by which the room-temperature hardness of high-aluminium compositions is less sensitive to changes in ageing temperature than that of compositions of low aluminium content.

Very large particle sizes can be readily developed by slow cooling from above the solution temperature.

An optimum-mean-free path exists between γ' particles and results in minimum creep rate. This optimum-mean-free path (together with the corresponding particle size for a given volume percentage of γ') varies with the applied stress.

Manganese and titanium expand the lattice of the γ matrix, and manganese reduces the quantity of γ' precipitate.

Electrolytic Polishing of Nickel-base Heat-Resisting Alloys

G. HÄUSSLER and V. PÄTZOLD: 'Studies of Electrolytic Polishing of Nickel-base Heat-Resisting Alloys.'

Neue Hütte, 1960, vol. 5, Nov., pp. 665–74.

In the introductory section of the paper, the authors present the results of their exploratory work on the electropolishing of nickel and chromium, which was carried out to supplement the findings of a literature review of the electrochemical processes involved in electropolishing. Details are then given of electrolytic-polishing experiments on four nickel-chromium-aluminium-titanium alloys which revealed the feasibility of applying the technique to this type of heat-resisting material. Optimum results were obtained with periodic polishing.

Electropolishing is shown to offer advantages over conventional hand polishing and etching, but it is emphasized that no universally applicable procedure can be laid down for nickel-base alloys as a class, since polishing conditions are governed by the composition and condition of the specific alloy.

Influence of Section Size on the Creep-Rupture Behaviour of Nickel Specimens Tested in Air

T. C. REUTHER, P. SHAHINIAN and M. R. ACHTER: 'Metals at High Temperature.'

Report of NRL Progress, 1960, Dec., pp. 25-6.

Tapered nickel creep specimens tested in air at 1500°F. (815°C.) have been shown to fracture in progressively larger sections of the taper as rupture life increases (i.e., as the stress is lowered). In previous investigations it was found that in air at 1500°F. (815°C.) nickel may exhibit a rupture life, at low stress levels, two orders of magnitude greater than that of specimens tested *in vacuo*, and, on the assumption that this pronounced oxidation strengthening might be more effective in the smaller- than in the larger-diameter sections of the taper (resulting in the observed shift of fracture position), the tests now noted were initiated to examine the suggested size effect during oxidation strengthening.

Creep-rupture tests, in which specimens of 0·25- and 0·375-in. (6·25- and 9·375-mm.) diameter were pulled to rupture in air at 1500°F. (815°C.), demonstrated that, at each of the stresses applied, the larger specimen broke sooner and had a higher minimum creep rate. At a stress of 2600 p.s.i. the smaller specimen exhibited pronounced oxidation strengthening, while the other did not: in the larger specimen this state of strengthening was achieved at a stress of 2500 p.s.i. It is concluded therefore that a size effect does exist.

Metallographic studies of the pattern and character of the cracks in fractured specimens and specimens taken from interrupted tests revealed that fracture generally occurred in sections containing extensive oxide-free (internal) cracks, while in the smaller sections of the taper all the cracks contained oxide. Interrupted tests showed that cracking begins at the minimum cross section and progresses towards heavier sections during the life of the specimen: i.e., a specimen interrupted at 300 hours revealed no cracks at the section where rupture ultimately occurred at 350 hours. During the early stages of specimen life, oxide-free cracks existed in the smaller sections of the taper, but these cracks became oxidized (and hence strengthened) before they could lead to rupture.

Metallography of Aluminium-Nickel Alloys

R. K. HART and M. J. HEYDUK: 'Metallography of Aluminium and Some Aluminium-1 w/o Nickel Alloys.'

U.S. Atomic Energy Commission, Argonne National Laboratory, Report ANL-6144, June 1960; 26 pp.

The study described was undertaken as a contribution to the understanding of the superior corrosion behaviour (*vis à vis* that of other aluminium alloys) of an aluminium-1 wt. per cent. nickel alloy in high-temperature water: its main aim was the development of a suitable method of preparing the surface of such alloys for, in particular, subsequent metallographic study of the second-phase precipitates.

Specimens of four materials were studied: high-purity aluminium; 'X8001' alloy (nickel 0·89, iron 0·48, copper 0·13, silicon 0·11, titanium 0·02, manganese 0·01, per cent., remainder aluminium); 'A255' alloy (high-purity aluminium containing 1·0 per cent. nickel and 0·1 per cent. titanium); 'A300' (produced by adding 1 wt. per cent. nickel to super-purity aluminium stock). To ensure a fine distribution of the second phase throughout the aluminium matrix, all the melts (with the exception of 'A300' control specimens, which were prepared in the as-cast state) were chill-cast and then heavily cold-rolled. Microstructures were examined by optical and electron-optical techniques.

The surface structure of the alloys was found to be equally well depicted on both mechanically and electropolished specimens. Because of the difficulties and time involved in mechanical polishing such soft materials, however, the electropolishing technique is preferred. Optimum results were obtained when the surfaces were abraded to at least 6 grade diamond before electropolishing.

The particulate material in the alloys was revealed with a high degree of fidelity by etching with either hydrofluoric acid or sodium hydroxide (it is also shown that cathodic etching is suitable for use with aluminium-nickel alloys, providing certain precautions are taken). Sulphuric acid was found unsuitable as an etchant, due to the formation of etch pits.

The results of the metallographic study are reported in detail, and the authors' comments are supplemented by numerous optical and electron micrographs. Particular attention is paid to anomalous furrow structures observed in electron micrographs.

Corrosion of Nickel-containing Aluminium Alloys in High-Pressure Steam

J. N. WANKLYN and N. J. M. WILKINS: 'Further Observations on the Uniform Corrosion of Aluminium Alloys in High-Pressure Steam.'

Jnl. Inst. Metals, 1961, vol. 89, Apr., pp. 289-91.

In an earlier paper the authors give details of an investigation of the corrosion of the nickel-containing aluminium alloy 'Aereal 1' in steam at temperatures in the range 270°-500°C. (see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 3, p. 58-9). Two main observations were made with respect to uniform corrosion: (1) At 325°C., the reaction with superheated steam followed a parabolic relationship with time up to at least seven days. The weight-gain at

constant time was proportional to the fourth power of the steam pressure, and at high pressures it approached, without apparent discontinuity, values found in saturated steam. (2) Other experiments suggested that, at a given pressure, the actual weight gains decreased with increasing temperature, and that the pressure-dependence was less at both 270°C. and 350°C. than at 325°C.

In the study now reported, sheet specimens of two alloys were exposed to steam at 325°C. and 1,000 p.s.i.: sintered aluminium powder (containing oxide 6·5, nickel 0·75, iron 0·75 and silicon 0·07, wt. per cent.); 'Aereal 1' (containing nickel 2·59, iron 0·43 and silicon 0·1, wt. per cent.). Before exposure, the specimens were cleaned by immersion for 10 minutes in 5 per cent. NaOH solution followed by 10 minutes in 5 per cent. HNO_3 solution, and then washed thoroughly and dried. Tests were carried out both in replenished and static steam.

Data on gain in weight are tabulated. Within the limits of the data presented, the authors conclude that: (1) the amount of corrosion observed in the present experiments was far lower than would be expected on the basis of previous work, and (2) under nominally similar conditions of steam temperature and pressure, considerable variations were observed in the degree of corrosion suffered by the specimens. These observations must, it is considered, be related to some property of steam other than temperature and pressure. The fact that the experiments in replenished steam differed from earlier tests only in that there was a constant flow of steam, and the apparent dependence of corrosion on steam flow, led to consideration of the possibility of there being a 'dryness' factor for the steam. Although the data presented give more weight to dryness than to pressure as a factor controlling corrosion, it nevertheless is deemed to remain true that the close adherence of the earlier results to a parabolic time law implies that the crucial changes are produced throughout the thickness of the oxide films, i.e., the effects of 'dryness' are not to be attributed to an influence on surface reaction rates. It is considered most likely that, as postulated earlier for pressure effects, variations in 'dryness' change the proportion of —OH groups in the oxide film, an effect which can influence ionic mobilities and, accordingly, rates of film growth. The concept of 'dryness' in steam corrosion is as yet speculative, but it is felt that the preliminary data reported are significant enough to have relevance for those working on the steam corrosion of aluminium, and possibly also other metals.

Behaviour of Nickel-containing Aluminium Alloys in High-Temperature Water

D. F. MACLENNAN: 'Corrosion-Product Films Formed on Aluminium in High-Temperature Water.' *Corrosion*, 1961, vol. 17, Apr., pp. 181t–4t.

Interest in the corrosion of aluminium in high-temperature water has arisen in connexion with its use in water-cooled nuclear-power reactors. Efforts to prevent the rapid corrosion to which the common aluminium alloys are subject at temperatures above about 200°C. resulted in the finding, by DRALEY and RUTHER (*ibid.*, 1956, vol. 12, Oct., pp. 480t–490t), that small additions of elements cathodic to aluminium, such as iron and nickel, changed the localized rapid attack to a uniform surface corrosion. KRENZ developed two aluminium-nickel-iron-silicon alloys (see below) which behaved well in water at 300°C.

Alloy	Ni %	Fe %	Si %
'155'	0·5	0·5	0·2
'157'	2·0	0·5	0·2

After a period of comparatively rapid attack, corrosion of these two alloys slows and proceeds at a linear rate, the '157 alloy' behaving slightly better than the '155'. The work now described was initiated in an attempt to contribute to the understanding of the mechanism by which alloying elements improve corrosion-resistance.

Samples of pure aluminium, 2S aluminium (99 per cent. purity), and the '155' and '157' alloys were exposed, in stainless-steel autoclaves, to water at 300°C. for short periods of time, and the corrosion product was subsequently examined by optical and electron microscopy (attention being focused on the initial stages of reaction). Micrographs illustrating the outer and inner surfaces of the oxide films formed are included in the paper.

The surface of all the specimens studied became coated with oxide almost immediately on exposure. It was found that the coating on the pure-aluminium specimens continued to grow until it thickened and cracked due to internal stress. In the case of the '157' alloy, intensified corrosion occurred around the second-phase particles, altering the corrosion product. It is suggested that corrosion-resistance is associated with the distribution of the second-phase particles in the alloys (the alloy exhibiting the highest corrosion-resistance contained the most uniform distribution of cathodic second-phase particles and the corrosion-product film contained a corresponding distribution of irregularities) and that the second-phase particles modify the film in such a way as to increase its protective qualities.

Corrosion of the '155' alloy occurred in a similar manner to that of the '157' alloy, except that the distribution of irregularities was less uniform and therefore less effective. The irregularities in the film of the '2S' aluminium specimens were large and far apart, and any beneficial effect on the film was therefore small.

Influence of Nickel on the Chromium and Carbon Relationship in Refining Stainless Steel

A. SIMKOVICH and C. W. McCOY: 'The Effect of Nickel on the Chromium and Carbon Relationship in Stainless-Steel Refining.'

Trans. Metallurgical Soc., A.I.M.E., 1961, vol. 221, Apr., pp. 416–7.

In the introduction to this note the authors allude to the development, by HILTY *et al.*, of the relationship between chromium, carbon and temperature during the oxidation period in melting plain-chromium stainless steel. The simplified version of the relationship is given by the equation

$$\log \frac{\%Cr}{\%C} = \frac{-13{,}800}{T} + 8{\cdot}76,$$

where the equilibrium concentration of carbon and chromium in the liquid metal at oxygen saturation is expressed in weight per cent., and T is the absolute temperature of the bath in degrees Kelvin. Although this equation was derived from data obtained from experiments restricted to plain-chromium steels, it is often applied to stainless steels containing nickel. It has, however, been speculated that high concentrations of nickel in steel would raise the carbon activity coefficient, and, on this basis, some variation in the equation might be expected when nickel is present in the bath. The study now described was undertaken to establish a quantitative correction which would account for the effect of nickel.

The data derived from these experiments gave rise to the following modified form of Hilty's equation which incorporates the necessary correction for nickel:

$$\log \frac{\%Cr}{\%C} = \frac{-13{,}800}{T + 4{\cdot}21\,[\%Ni]} + 8{\cdot}76.$$

Creep and Creep-Rupture Relationships in 18-8-Mo Austenitic Steel

F. GAROFALO, R. W. WHITMORE, W. F. DOMIS and F. VON GEMMINGEN: 'Creep and Creep-Rupture Relationships in an Austenitic Stainless Steel.'

Trans. Metallurgical Soc., A.I.M.E., 1961, vol. 221, Apr., pp. 310–19.

Previous work on a variety of metals and alloys tested at elevated temperature under creep conditions has shown that the empirical relationship

$$t_r = \frac{C}{(\dot{\varepsilon}_m)^{\alpha}}$$

exists between rupture life, t_r, and minimum creep rate, $\dot{\varepsilon}_m$. This relationship, in which C and α are, in some cases, independent of stress or temperature, indicates, in a general way, that creep rupture depends on creep behaviour prior to the tertiary creep stage. A more detailed determination of the factors which control creep rupture requires, therefore, more knowledge of the relationship between rupture and creep behaviour. The studies now reported were initiated to obtain such information, and were designed, on a statistical basis, to establish the variability of the various creep properties measured, and to determine empirically the stress dependence of minimum creep rate and rupture life.

Type 316* austenitic stainless steel was subjected to constant-load creep-rupture tests at 1100°, 1300° and 1500°F. (595°, 705° and 815°C). The data obtained, which are presented *in extenso*, are deemed to warrant the follow conclusions:

'(1) At any stress level at the three test temperatures a measurable amount of scatter is found in the minimum creep rate and rupture life. This is particularly true at 1100°F. (595°C.). The spread in minimum creep rate and rupture time is essentially the same at 1300°F. (705°C.) as at 1500°F. (815°C.).

'(2) The empirical relation,

$$t_r = \frac{C}{(\dot{\varepsilon}_m)^{\alpha}},$$

between rupture life, t_r, and minimum creep rate $\dot{\varepsilon}_m$, is substantiated within certain limits of temperature and stress. It is found that α is nearly unity, as reported by MONKMAN and GRANT (*Proc. Amer. Soc. Testing Materials*, 1956, vol. 56, p. 834). It is also found that,

$$C = A\,(\varepsilon_2 - \varepsilon_1),$$

where A is independent of stress at constant temperature but increases somewhat with temperature, and where $(\varepsilon_2 - \varepsilon_1)$ is the secondary creep strain. For the material tested in this study, the breaks in the log σ_0–log t_r plots, where σ_0 is the initial stress, are traced to either a deviation from linearity at high stresses in the log σ_0–log $\dot{\varepsilon}_m$ plot, or to a decrease in the amount of secondary creep strain.

'(3) Secondary creep strain is found to depend on the type of grain-boundary precipitate, which affects grain-boundary migration. At 1100°F. (595°C.), a fine and continuous precipitate is found at grain boundaries, with no evidence of grain-boundary migration. At this temperature, $\varepsilon_2 - \varepsilon_1$, remains essentially constant. Precipitation of large discrete particles at grain boundaries by heating to 1500°F. (815°C.) for 24 hours before testing at 1100°F. (595°C.) shows no systematic change in $\dot{\varepsilon}_m$, but secondary creep strain and rupture life are measurably increased, and grain-boundary migration is observed. At 1300°F. (705°C.) and 1500°F. (815°C.), secondary creep strain up to rupture lives of 1000 and 300 hours, respectively, is appreciably greater than at 1100°F. (595°C.). For longer rupture lives, corresponding to lower stresses, secondary creep strain decreases markedly. This drop is believed to be caused by the greater predominancy of grain-boundary shearing as the stress is diminished. At these two temperatures grain-boundary migration is observed at all stress levels employed.'

* Composition: carbon 0·07, manganese 1·94, silicon 0·38, sulphur 0·021, phosphorus 0·01, aluminium 0·003, nitrogen 0·043, chromium 18, nickel 11·4, molybdenum 2·15, per cent.

Influence of Trace Amounts of Boron on the Creep Properties of Austenitic Steels

L. COLOMBIER: Letter to Editor.

Revue de Métallurgie, 1960, vol. 57, Dec., pp. 1105–6.

Following the finding that the creep properties of chromium-nickel steels of 18–8–Ti type melted in an acid-lined induction furnace were superior to those of similar types of steel produced in furnaces with basic lining, the author investigated possible reasons for this effect, and established that trace amounts of boron had been introduced into the former steels *viâ* the acid refractory. In view of the known beneficial effects of boron on the high-temperature properties of 'superalloys', a test programme was initiated to determine whether the improvement in the properties of the acid-melted steels was attributable to the presence of boron.

Stress-rupture tests were carried out on specimens of 18–8–Ti and 18–8–Nb stainless steels (1) free from boron, and (2) containing up to 0·0033 per cent. of boron. The data obtained, which are presented in this note, confirm the previously observed improvement attained in the creep properties of steels in the presence of boron.

Influence of Minor Alloying Additions on the High-Temperature Mechanical Properties of Cast Chromium-Nickel-Molybdenum Heat-Resisting Stainless Steels.

J. SALVAGGI: 'Final Report on Evaluation of Cast Alloys for Use at High Temperatures.'

U.S. Navy, Bureau of Ships Report KB-1137-M-4, 1957; 11 pp.

In previous work at Cornell Aeronautical Laboratories, Inc. (concerned with the evolution of heat-resisting materials 'leaner' in alloying constituents than those commercially available), a study of the influence of boron on the high-temperature properties of wrought austenitic steels resulted in significant improvements in the properties of Type 316 18–12–Mo steel. The work now described was carried out to investigate the feasibility of achieving the same effect in cast steels of similar compositions.

The eight heats of austenitic steel produced for study (containing carbon 0·27–0·48, chromium 17–20, nickel 12–20, molybdenum 2·5–5, per cent.) were modified by minor additions of niobium, niobium + boron, niobium + titanium + boron + tungsten, titanium + boron + tungsten, niobium + boron + tungsten, or boron + tungsten. As-cast specimens were subjected to tensile tests, and to rupture tests at 1500°F. (815°C.) and, in some cases, at 1600°F. (870°C.). Two low-alloy chromium-molybdenum-niobium-boron-tungsten steels were also evaluated.

Alloying additions of niobium (*per se* and in combination with boron and tungsten) considerably improved the cleanliness and castability of the air-melted heats. Two steels of the following compositions exhibited excellent properties at 1500°F. (815°C.), and the 100-hour rupture-stress values obtained (in the region of 24,000 p.s.i. (10·5 t.s.i.; 17 kg./mm.²)) represented the highest strengths achieved by any of the cast compositions studied: (1) carbon 0·48, chromium 19·5, nickel 19·58, molybdenum 4·37, niobium 1·4, boron 0·49, tungsten 1·22; (2) carbon 0·45, chromium 20–26, nickel 20·64, molybdenum 4·66, boron 0·6, tungsten 2·84, per cent.

In the case of the two ferritic steels, 100-hour rupture-strength values at 1200°F. (650°C.) were comparable to those reported in the literature for several commercial stainless steels. Ductility was, however, extremely low, and would, it is considered, render the steels virtually unusable.

See also

Influence of Vacuum-Melting on the High-Temperature Mechanical Properties of a 20-20-5Mo Stainless Steel containing Minor Alloying Additions

F. J. GILLIG: 'Final Report on Improvement of Cr–Ni–Mo Iron-Base Alloys by Vacuum Melting and Casting.'

U.S. Navy, Bureau of Ships Report KA-1289-M-6, Sept. 30, 1959; 13 pp.

The test programme referred to in the previous abstract ended before further work could be carried out to ascertain whether the alloying constituents of the two most promising steels developed were in optimum combination. It was felt that minor changes in composition, and the use of vacuum-melting, might improve the properties of the steels still further. The study now reported was initiated to determine whether this would in fact be so, and, specifically, to investigate the effect of vacuum-melting and minor changes in composition on the properties of steels of 20Cr–20Ni–5Mo–Fe basis containing various combinations of niobium, tungsten, titanium, boron and aluminium.

Twenty-one heats were cast, and specimens were tested in the as-cast condition, using the 100-hour rupture stress at 1500°F. (815°C.) as the criterion for evaluation. The compositions selected as the most promising on this basis were then subjected to rupture tests at 1350°F. (735°C.) and 1600°F. (870°C.), to creep tests for 1000 hours at 1500°F. (815°C.), and to tensile tests at room temperature and 1500°F. Metallographic studies were carried out, and, in certain cases, response to heat-treatment was determined.

The most satisfactory results were obtained with a steel of the following composition: carbon 0·52, manganese 1, silicon 1, chromium 17·79, nickel 15·39, molybdenum 3·78, boron 0·5, tungsten 2·24, niobium 0·2, per cent. Tensile strength, yield strength and elongation at 1500°F. (815°C.) were, respectively: 46,700 p.s.i. (20·8 t.s.i.: 32·8 kg./mm.²), 34,800 p.s.i. (15·5 t.s.i.: 24·5 kg./mm.²), 7 per cent.

Rupture strengths for 1000 hours were:

°F.	°C.	p.s.i.	t.s.i.	kg./mm.²
1350	735	33,000	14·7	23·2
1500	815	19,500	8·7	13·7
1600	870	11,500	5·0	8·0

Although the steel derives its elevated-temperature strength from a precipitation-hardening mechanism, it requires no heat-treatment and it was not found possible to improve properties at 1500°F. (815°C.) by heat-treatment (though a stabilizing treatment may, it is considered, enhance properties for low-temperature applications). The steel exhibited excellent castability and may be air-melted. Oxidation resistance at 1500°F. (815°C.) was very good.

Influence of Temperature and Section Size on the Tensile Properties of 18-8 and 18-11-Nb Stainless Steel

W. H. PRYLE and E. T. WESSEL: 'Tensile Properties of A.I.S.I. Types 304 and 347 Stainless Steels at Moderate Temperatures for Section Sizes Ranging from Bars to Extremely Large Forgings.'

Amer. Soc. Mechanical Engineers, 1960, *Paper* 60-*WA*-9; 10 pp.

Current trends in design have focused attention on the need for precise data on the mechanical properties, in particular, yield strength, of the austenitic stainless steels (the A.S.M.E. Code permits designers to take advantage of a relatively large percentage of the yield strength). In certain cases, account must be taken of the possibility of localized yielding in the presence of high thermally induced stresses. It is important therefore to obtain information on the degree to which properties can be expected to vary over the relevant temperature range with different lots of a given type of steel and with differences in section size. The work now described was carried out, on A.I.S.I. Type 304 and 347 (i.e., 18-8 and 18-11-Nb) stainless steels, to obtain this information and to acquire reliable design data. At the time the investigation was initiated, few data were available for the temperature range of interest [room temperature to 800°F. (425°C.)], although data for temperatures higher than 1000°F. (540°C.) were extensive. Tensile properties, and particularly yield strength, were therefore determined in the temperature range 75°-800°F. (24°-425°C.).

The specimens tested were obtained from commercially produced stainless steels acquired, in the following section sizes, under equivalent purchase specifications calling for annealed material with a minimum yield strength of 30,000 p.s.i. (13·5 t.s.i.: 21 kg./mm.²) at room temperature: bar stock ranging from ¾-2 in. in diameter; intermediate-size forgings ranging from 9-12¾ in. in diameter; large forgings (the largest [in Type 347] was produced from a 33-ton ingot forged to a slab 83×92×21 in. thick; the other two forgings [in Type 304] were 46 in. in diameter and 14 in. thick). In the case of the forgings, a study was made of the effects of specimen location within the forging and specimen orientation with respect to the primary working directions.

The data derived from the test programme show that, in all cases, 'the effect of increased temperature was to decrease both strength and ductility. This effect prevailed for all size categories and is most pronounced in the range from 75° to 300°F. (25° to 150°C.).

'In any one size category a large difference of tensile properties, particularly yield strength and elongation, was observed between the various lots of a given type of steel. This difference remained constant over the entire temperature range that was studied. The variation in properties correlated quite well with variations in hardness. The primary source of these variations was traced to differences in the annealing treatments that were given to the various lots of steel. When the various lots of a given type of material were given a consistent re-annealing treatment, the variation in tensile properties was greatly reduced.

'Within any given lot of steel, the tensile properties were quite uniform. In the forgings it was possible to obtain samples from various locations. The properties were relatively consistent, independent of the location from which the samples were taken. There was one exception. In the large forgings there was a variation in ductility through the thickness of the forging. The central regions tended to exhibit poorer ductility than the surface regions. However, this difference only prevailed for specimens that were oriented such that their axis coincided with the thickness direction of the forging.

'At any given location in a forging, the tensile ductility was found to be dependent upon the orientation of the specimen, although the yield and ultimate strengths were not affected. In the one large forging in which all three orientations were investigated, it was found that the ductility of the specimens oriented in the thickness direction was about half of that of those oriented in either the transverse or longitudinal directions. However, notched-tensile and impact tests revealed that the poorer tensile ductility of this orientation did not lead to notch sensitivity or embrittlement problems.'

Occurrence of Ti_2S in Titanium-stabilized Austenitic Steels

K. WETZLAR and G. LENNARTZ: 'Occurrence of a Titanium Sulphide, Ti_2S, in Titanium-stabilized Austenitic Chromium-Nickel Steels.'

DEW Technische Berichte, 1961, vol. 1, Jan., pp. 15-16.

In earlier investigations of the phases present in titanium-stabilized chromium-nickel 18-8 stainless steels, X-ray examination of isolated residues revealed the presence not only of $Cr_{23}C_6$, TiC, TiN and σ phase, but also of an unidentified precipitate which occurred irrespective of the prior heat-treatment (see *Nickel Bulletin*, 1956, vol. 29, No. 8-9, pp. 172-3). In the present paper the authors conclude, on the

basis of X-ray and metallographic data, that the precipitate is a sulphide, Ti_2S, of hexagonal structure.

The presence of titanium sulphide in titanium-stabilized steels will, it is considered, have the effect of reducing (to a degree depending on the behaviour of the nitrogen present) the amount of titanium available for combination with carbon. Data on the composition of the sulphide enabled calculation of the amount of titanium combined with sulphur: at the usual sulphur contents of 0·015–0·02 per cent., the titanium percentages would be in the range 0·05–0·06.

Properties of Non-Magnetizable Steels

See abstract on p. 152.

Kinetics of Pitting Corrosion of Chromium-Nickel Stainless Steels

E. BRAUNS and W. SCHWENK: 'Kinetics of Pitting Corrosion of Passive Chromium-Nickel Steels.'

Werkstoffe u. Korrosion, 1961, vol. 12, Feb., pp. 74–80.

In the investigation reported, pitting corrosion in the presence of chlorine ions was studied in relation to six chromium-nickel stainless steels (of 18-10, 18-11-2Mo, 19-9-0·5Mo-Ti, 18-12-2Mo-Ti, 19-10-Nb and 18-12-2Mo-Nb type) exposed to a buffer solution containing the redox system Fe(II)/Fe(III). Other experiments were carried out in chloride-containing electrolytes free from redox systems. Measurement of the electrode potential and the electrode impedance provided information on the kinetics of pitting corrosion.

Potentiostatic measurements in electrolytes not containing a redox system showed that pitting corrosion occurs only in a specific potential region. In contrast to the findings relating to nitrate-free electrolytes, this region is limited to positive potentials in the presence of nitrate ions. The density of the pits (which did not vary with time), the induction period and the quasi-stationary corrosion current density were found to be potential-dependent functions, the curves for which followed a monotonous course in nitrate-free electrolytes, and, in nitrate-containing electrolytes, a symmetrical course to the limiting potentials, passing through an extreme value. Beyond the limiting potential, the number of pits and the corrosion current increased, and the induction period decreased. Apart from a few transition phenomena in the region of the limiting potentials, the corrosion current increased as a function of the square of time. Since the factor of this function of time is potential-dependent, the graph has an anodic inclination, an effect which agrees with the measurements carried out in the redox buffer solution.

The experimental results can, it is considered, be interpreted by assuming a chemisorption process as a starting reaction. Concerning the stable coexistence of active and passive regions, in connexion with the 'all-or-nothing' law of passivity, the authors assume a resistance polarization, as well as hydrochloric-acid concentration in the pits.

Nature of the Oxide Film Formed on 18-8-Ti Stainless Steel Immersed in Concentrated Hydrogen Peroxide

J. DIEDERICHSEN: 'The Nature of the Oxide Film Formed on Stainless Steel Immersed in Concentrated Hydrogen Peroxide (H.T.P.).'

Jnl. Applied Chemistry, 1960, vol. 10, Dec., pp. 497–505.

When stainless steel is immersed in H.T.P., it is generally found that the liquid decomposes at a faster rate when the surface of the metal has not been treated by previous immersion. The main object of the work reported (to study the stability of the surface condition attained by stainless steel immersed in H.T.P.) involved examination of the nature and rate of formation of the surface film and the influence of its formation on the rate of decomposition of the H.T.P.

In general, the H.T.P. employed in the experiments was of 85 per cent. w/w concentration (the stabilizer content being 4·6 mg. of tin per litre, and the pH 5·8). The stainless steel selected for investigation conformed to *B.S.* 970 *En58B**. Measurements were made of the potential of the steel in H.T.P., and the oxide films formed during immersion were studied by electrometric methods and chemical analysis. Tests were also carried out to determine the compatibility of stainless-steel and mild-steel specimens with H.T.P. during total immersion at 30°C.

On the basis of the results presented, the author concludes that the film produced on stainless steel during immersion in H.T.P. at 30°C. consists mainly of oxides of iron, and that it grows to a final thickness of about 3×10^{-6} cm. in three to four weeks. The comparatively thick oxide film formed is stable in air and in water for at least a week.

The compatibility characteristics of stainless steel are explained by the fact that iron oxide is practically insoluble, and is not catalytic, in H.T.P. of the usual pH, and remains as a film on the surface of the steel. Chromium is highly catalytic, and, together with nickel, passes into solution: the increased rate of decomposition of the H.T.P. is caused mainly by dissolved chromium. If the stainless steel on which the iron-oxide film has been formed is transferred to a new charge of H.T.P., the rate of solution of chromium is slower than before (because the ions have to diffuse through the oxide film), and, consequently, the rate of decomposition of H.T.P. is also lower. The compatibility of the steel with fresh H.T.P. can be improved until the maximum thickness of oxide film is formed (in approximately four weeks at 30°C.), and, in the case of steel with this maximum thickness of oxide film, the rate of decomposition increases during storage because the chromium is still diffusing slowly through the oxide film into the H.T.P.

* Chromium 17-20, nickel 7-10, carbon $\leqslant$ 0·15, titanium $\geqslant$ 4 × carbon, per cent.

Corrosion of Nickel-containing Materials by Formic, Acetic and Phosphoric Acids under Heat-Transfer Conditions.

N. D. GROVES, C. M. EISENBROWN and L. R. SCHARFSTEIN: 'Corrosion of Metals by Weak Acids under Heat-Transfer Conditions.'

Corrosion, 1961, vol. 17, Apr., pp. 173t–6t.

Since very few corrosion data are available on the corrosion of materials of construction for chemical plant under conditions in which the temperature of the alloy or steel is higher than that of a boiling acid solution, the authors undertook the investigation reported to obtain information on the influence of temperature on the corrosion behaviour of representative materials in boiling solutions of three common acids.

The following six materials were selected for study, as exhibiting important compositional differences representative of those found in alloys and steels employed in the chemical process industry: 18–12–Mo and low-carbon 18–8 chromium-nickel stainless steels (Types 316 and 304L), the 30–20 chromium-nickel-molybdenum-copper-niobium steel 'Carpenter 20Cb', the nickel-molybdenum-iron-base alloy 'Hastelloy B', the nickel-chromium-iron alloy 'Inconel', and the nickel-copper alloy 'Monel'. Formic, acetic and phosphoric acids were chosen as corrosive media.

Test specimens were exposed to three concentrations of each acid (10 per cent., 50 per cent. and concentrated) at metal temperatures of 230°, 257° and 284°F. (110°, 125° and 141°C.). With the exception of the concentrated phosphoric acid (which boiled at temperatures above the limit of the test apparatus, and was maintained at approximately 210°F. [100°C.] during exposure both with and without heat transfer), the effects of each acid were evaluated at its atmospheric boiling point. The tests were continued for 48 or 96 hours, and a special heat-transfer device was employed to obtain data permitting comparison of corrosion rates obtained with and without heat transfer.

On the basis of the corrosion data derived from the tests (corrosion rates are summarized in tables covering each material and test temperature), the authors conclude that, of the materials studied, 'Carpenter 20 Cb' and 'Hastelloy B' most consistently showed the highest corrosion-resistance, irrespective of the metal temperature: corrosion rates did not differ significantly under heat-transfer conditions. 'Monel' exhibited the highest sensitivity to the effects of heat transfer.

Corrosion rates with formic acid tended to increase with rise in metal temperature, while acetic acid exhibited the least tendency in this respect.

The authors' overall conclusion is that corrosion rates obtained by extrapolation of simple immersion data to higher metal temperatures may be seriously erroneous.

Resistance of High-Alloy Steels to Corrosion by Nitric Acid

J. BÜNGER: 'Corrosion of High-Alloy Steels by Nitric Acid. II.'

Werkstoffe u. Korrosion, 1961, vol. 12, Mar., pp. 141–8.

The author's earlier investigations had revealed the corrosion behaviour of high-alloy steels in nitric acid to be considerably influenced by the carbon and chromium contents of the steel (*ibid.*, 1958, vol. 9, Dec., pp. 747–55: see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 4, pp. 120–1). Since the corrosion-resistance of 18–8-type chromium-nickel steels does not, under certain conditions of temperature and acid concentration, meet service requirements, it was deemed of value to extend the scope of the corrosion tests to include steels of higher chromium content. Steels with 28–30 per cent. chromium exhibit good resistance to nitric acid, and the results of the previous study indicated that their tendency to embrittlement can be counteracted, without too great an effect on corrosion-resistance, by rendering them austenitic by addition of nickel. The behaviour of the abrasion-resistant high-chromium high-carbon steels was also of interest in this respect, since their chromium content might be expected to confer a certain degree of corrosion-resistance. The compositions of the steels selected for study in the light of these considerations are given in the table on p. 166.

In the corrosion tests reported, the specimens were exposed, at various temperatures, to concentrations of chemically pure nitric acid ranging (generally at intervals of 10 per cent.) from 1 to 67 per cent., and maintained at temperatures varying, at intervals of 20C.°, up to the boiling points of the solutions. Tests at boiling point lasted for 24 hours; at other temperatures they were continued for 7 days. Weight losses for each steel, converted into corrosion rates, were plotted as a function of temperature and acid concentration. The iso-corrosion diagrams so obtained are presented and discussed in the paper. Data derived from tests with boiling nitric acid at concentrations of 30, 40, 50, 60 and 67 per cent. are used to give a graphical evaluation of the influence of chromium and carbon contents on the corrosion rate. Corrosion rates obtained for specimens subjected to tests in boiling acid are reproduced in the table on p. 166.

The iso-corrosion diagrams presented in the paper permit determination of the corrosion rates of a specific steel under given conditions of acid concentration and temperature. Of the steels studied, that containing carbon 0·1, chromium 30, nickel 20, per cent., proved the most corrosion-resistant: the same steel exhibited no signs of intergranular corrosion when exposed to 50 and 57 per cent. boiling nitric acid after heat-treatment at 680°C.

When the data obtained from the tests carried out in the present and previous study were plotted to reveal the relationship between the corrosion rate (on a logarithmic scale) and carbon and chromium contents

Corrosion of High-Alloy Steels in Nitric Acid

(*See abstract on p.* 165)

Composition							Corrosion Rates in Boiling Nitric Acid, mm./year				
C %	Si %	Mn %	Cr %	Ni %	Mo %	Cu %	30 %	40 %	50 %	60 %	67 %
1·63	0·53	0·49	25·20	0·32	0·10	0·05	1·54	2·22	4·27	—	—
1·64	1·45	0·40	29·45	0·34	0·10	0·00	2·08	—	6·81	—	—
0·91	1·03	0·67	27·30	0·51	0·10	0·00	0·18	0·47	1·22	2·06	2·52
0·07	0·78	0·43	26·25	0·36	0·11	0·03	0·019	0·042	0·12	0·18	0·24
0·23	1·08	0·62	24·85	9·10	0·34	0·06	0·021	0·21	0·22	0·50	0·52
0·13	2·04	0·80	24·50	20·10	0·08	0·05	0·019	0·042	0·17	0·46	0·64
0·06	1·09	1·08	24·45	19·80	0·10	0·00	0·023	0·030	0·085	0·14	0·19
0·09	0·84	0·88	29·65	19·30	0·13	0·00	0·009	0·021	0·050	0·12	0·16
0·12	0·44	0·33	13·10	0·56	0·13	0·08	0·67	1·12	3·45	4·63	6·00
0·20	0·53	0·32	13·15	0·62	0·23	0·00	0·70	1·25	2·60	—	—
0·43	0·53	0·49	12·35	0·35	0·06	0·06	3·40	7·10	17·0	—	—
0·10	0·86	0·36	16·70	0·36	0·08	0·15	0·25	0·45	0·74	1·08	1·22
0·23	0·57	0·24	16·25	1·60	0·02	0·04	0·45	0·75	1·30	—	—
0·40	0·40	0·35	16·20	0·70	0·98	0·10	0·69	1·16	1·68	2·64	3·35
0·88	0·57	0·32	17·20	0·26	1·19	0·00	1·20	4·20	6·00	—	—
0·08	0·92	0·62	22·10	1·16	0·10	0·15	0·12	0·28	0·40	—	—
0·11	0·88	1·02	19·65	2·30	0·02	0·00	0·16	0·36	0·60	—	—

(on a decimal scale), the graphs were in the form of straight lines: i.e., the influence of both elements may be expressed as a parabola, rising with concentration in the case of the carbon and falling in the case of the chromium. In assessing the chromium content associated with corrosion-resistance, the percentage of the metal combined with carbon (amounting, on the basis of the carbide Cr_3C, to $13 \times \%C$) must be deducted from the total content.

High nickel contents (20 per cent.) had no noticeable effect on the degree of corrosion, a result which indicates that, from the point of view of corrosion, it is immaterial whether the structure is ferritic or austenitic. The essential constituent is, it is concluded, the free-chromium content, the effect of which is governed by a parabolic law.

In plotting the data as a function of carbon content, three parabolas are obtained, applying, respectively, to chromium contents of 13, 17 and 25 per cent. At carbon contents less than 0·1 per cent., corrosion-resistance is governed exclusively by the chromium content.

Corrosion of 25-20 Austenitic Steel by Synthetic Fuel Oil Ash

H. L. LOGAN: 'Elevated-Temperature Corrosion of Type 310 Stainless Steel by Vanadium Compounds.' *Corrosion*, 1961, vol. 17, Apr., pp. 185t–7t.

The test programme described formed part of an investigation of the corrosion of A.I.S.I. Type 310 stainless steel* by fuel-oil ash in progress at the National Bureau of Standards. The results of the first phase of the study, that concerned with determination of the minimum temperature at which synthetic oil ash would attack the steel, has been previously reported in a paper (*ibid.*, 1959, vol. 15, Aug., pp. 443t–6t: see abstract in *Nickel Bulletin*, 1959, vol. 32, No. 11, pp. 338–9) which also contained some data derived from a study of the mechanism involved in fuel-oil-ash corrosion of the steel. In

* Composition: carbon 0·06, manganese 1·53, silicon 0·19, phosphorus 0·022, sulphur 0·005, chromium 24·8, nickel 21·2, per cent.

the present paper the author summarizes the data obtained on the mechanism of attack since publication of the earlier report.

The synthetic oil-ash mixture selected for study was placed in a tube fabricated from Type 310 steel. The tube was then sealed and subjected to temperatures in the range 1700°–2000°F. (925°–1095°C.) for from 6 to 200 hours. An atmosphere of wet or dry air or helium was introduced into the tube during testing. Some of the tubes were, prior to testing, heated in air to produce heavy oxide films on the interior surface. The synthetic ash mixture employed in most of the work reported comprised 67 wt. per cent. of vanadium pentoxide and 33 wt. per cent. of sodium vanadate, a mixture which contained a little less vanadium pentoxide than the compound $Na_2O \cdot V_2O_4 \cdot 5V_2O_5$ reported to be the most corrosive of any synthetic ash mixture made up of V_2O_5 and $NaVO_3$. At the minimum temperature determined for attack on the steel (1075°F.: 580°C.), the mixture would consist of solid $Na_2O \cdot V_2O_4 \cdot 5V_2O_5$ and liquid $NaVO_3$, but at the temperature at which most of the data were obtained the mixture was entirely molten and highly viscous.

The conclusions drawn from the results presented are quoted below:

'1. Catastrophic failures of Type 310 stainless-steel specimen tubes have occurred under conditions where moist air was introduced into the tube containing 67 wt. per cent. V_2O_5 and 33 wt. per cent. $NaVO_3$ at 1900–2000°F. (1035°–1095°C.), providing the specimen had previously been oxidized.

'2. No catastrophic failures occurred in specimens exposed under very similar conditions except that atmospheres of dry air or moist helium were introduced into the tubes instead of moist air.

'3. Grooving was found in sections of specimens adjacent to regions where failure had occurred. Grooving was observed in regions where there had been grain growth. In some instances, material of a second phase was found at the root of a groove penetrating into the steel along grain boundaries.

'4. Using the technique of leaching out various components, the slag produced in a catastrophic failure of a specimen was found, by X-ray-fluorescent and -diffraction techniques, to contain gamma Fe_2O_3, Cr_2O_3, NiO, V_2O_3 and an unidentified vanadate. The lattice parameter of the Fe_2O_3 was $a = 8{\cdot}326$ Å.

'5. Chemical analysis of the slag from a tube that had failed catastrophically indicated that the chromium-iron and manganese-iron ratios were the same in the slag as in the basis steel. The nickel-iron ratio in the slag was ·432, compared to ·406 in the basis steel. This would suggest the possibility that the nickel was selectively absorbed by the slag prior to general attack.'

Corrosion of Superheaters and Reheaters in Pulverized-Coal-Fired Boilers

C. CAIN and W. NELSON: 'Corrosion of Superheaters and Reheaters of Pulverized-Coal-Fired Boilers. II.' *Amer. Soc. Mechanical Engineers*, 1960, *Paper* 60-*WA*-180; 7 pp.

In a previous paper, the authors presented the results of laboratory and field studies of high-temperature fireside corrosion of reheater and finishing superheater tubes in modern pulverized-coal-fired boilers (see abstract in *Nickel Bulletin*, 1961, vol. 34, No. 1, pp. 16–17). Complex iron and/or aluminium alkali sulphates were shown to form in fireside ash deposits and to become corrosive when molten on tubes at steam temperatures higher than 950°F. (510°C.). It was concluded that molten compounds migrate through the ash deposit to the tube surface (as a result of the thermal gradient existing in the deposit) and react with the metal according to the following equation:

$3Fe$	$+$	$SO_4^=$	$\rightarrow$	Fe_3O_4	$+$	$S^=$
Tube metal		Molten complex sulphate		Iron oxide		Sulphide

The skin temperature for this rapid liquid-phase attack [in the region of 1025°–1300°F. (550°–705°C.)] was delimited by (1) the melting point of the mixture of complex alkali sulphates present, and (2) their thermal-stability limit. The presence of ash deposits at a metal temperature within these limits was found to be necessary for occurrence of rapid corrosion by this mechanism. Stainless-steel shields attached to the tube surfaces effectively prevented corrosion (due to the fact that the skin temperature of the shields was above the temperature limit for formation of the corrosive compounds). Studies of the corrosion process have continued, and the data obtained to date are discussed in the present paper.

The results, derived from tests on T9 (9 per cent. chromium-molybdenum), T22 (low-alloy chromium-molybdenum), TP304 (18–8 chromium-nickel) and TP 430 (16 per cent. chromium) steels, are discussed in sections relating to factors affecting the rate and temperature range of liquid-phase corrosion, the use of stainless-steel shields as a preventive measure, and detection of liquid-phase attack. The characteristics of coal-ash and residual-oil-ash corrosion are compared.

The temperature range for rapid liquid-phase corrosion of reheaters and superheaters by molten complex sulphates is, it is concluded, a function of the species and proportion of complex sulphates present. The upper limit is relatively constant at about 1300°F. (705°C.), but the lower limit may vary considerably. Sulphidation and carburization of the metal structure increase the corrosion rate, and all the alloys tested were subject, in some degree, to liquid-phase attack.

Shielding of surfaces susceptible to corrosion is regarded as the most effective preventive measure

yet developed, but the material used for the shields was found to be a vital factor in this respect. At one installation the lead pendent-outlet reheater tubes of a unit were shielded with Type 430 steel. The shields were fabricated and installed in exactly the same way as Type 304 18–8 shields incorporated in other units. After one year's operation the ferritic-steel shields, having almost completely disintegrated, were replaced with TP304 shields, which at the time of publication of the paper had been in service for about a year without appreciable ash build-up or damage: a finding which indicates that use of an austenitic rather than a ferritic steel is necessary for corrosion-resistance.

The data show that liquid-phase corrosion of austenitic steels may be detected by inspection of the tube surfaces for signs of pitting or notching, or by metallographic examination for evidence of penetration of sulphides. In the case of ferritic steels, determination of the sulphide content of the scale overlaying corroded areas, or use of a sulphur-print technique, distinguishes between liquid and gas-phase corrosion. The potential corrosiveness of a deposit may be estimated by determining the acid content of a water solution of the inner corrosive layer adjacent to the tube surface.

Service Performance of Stainless-Steel Condenser Tubes

R. H. PELL: 'Performance of Stainless-Steel Condenser Tubes.'

Amer. Soc. Mechanical Engineers, 1960, *Paper* 60-*WA*-217; 3 pp.

Unit No. 6 of the Monongahela Power Company's Rivesville Power Station came into service in 1951 with a capacity of 90,000 kw. Steam conditions at the throttle are 1250 lb. and 950°F. (510°C.). The Monongahela River, which furnishes the station with cooling water, is highly corrosive due to coal-mine drainage; its pH ranges from less than 3 during dry seasons to 6 during rainy seasons. Fouling and severe corrosion of the 88–10–2 copper-alloy condenser tubes proved a continuous problem, and it was difficult to maintain an acceptable state of cleanliness, since adequate cleaning exposed the bare metal to acid attack, with subsequent formation of new corrosive films. The copper-alloy tubes tended quickly to acquire a rough surface coated with a tenacious layer of corrosion product on the water side, and it was also noted that the steam side became coated with a heavy film of iron oxide. The life of the tubes in the Unit No. 6 condenser was about 6⅔ years. In an attempt to discover a more suitable material of construction from the point of view of performance and life expectancy, the Company has, over the years, tested a number of copper alloys and several types of stainless steel. Tubing in Type 304 18–10 stainless steel, installed in one of the older condensers in 1937, showed no measurable attack when the alloy tubing was replaced in 1945. Examination of the same tubing in 1954 revealed that, after 17 years' service, no pitting or measurable decrease in wall thickness had occurred. At that time the use of Type 304 steel was ruled out because of its higher cost and reputedly poorer heat-transfer characteristics, but subsequently the feasibility of employing stainless steel in retubing the No. 6 condenser was given close attention.

Since many factors, such as degree of cleanliness, influence heat-transfer characteristics, it was believed that the actual thermal conductivity of the steel might be of lesser importance in evaluating its suitability than previously thought, and it seemed reasonable to assume that the greater resistance of the stainless steel to oxidation and fouling would offset some of the differences in thermal conductivity. Moreover use of stainless steel would, it was considered, enable the wall thickness of the tubing to be reduced, with consequent reduction in costs and improvement in heat-transfer characteristics. The selection of welded-seam 22Bwg Type 304 stainless-steel tubing for evaluation was influenced by the following factors: a life expectancy of 30 years for the stainless steel, compared with 7 years for the copper; welded stainless-steel tubes were available in a reasonable price range; satisfactory results were obtained with 22Bwg tubing when expanded into the tube sheet; experience indicated that stainless steel could be used in conjuction with Naval brass tube sheet without any detrimental effects from galvanic attack. In July 1958 the surface condenser of No. 6 Unit was therefore completely re-tubed with 9,234 welded tubes in Type 304 stainless steel. This paper gives details of the performance of the tubing during the two years since its installation.

On the basis of the information given, the author concludes that stainless-steel condenser tubes are capable of 'much better heat transfer than expected. The excellent corrosion-resistance ensures a long service life, in contrast to our former experience. The hard stainless surface resists fouling and corrosion, and cleanliness is more easily maintained. The resistance to wear and erosion permits the use of thinner-wall tubes. Additional benefit is received because of its tendency for self-cleaning and natural scouring. Where fouling and corrosion are a problem, as at Rivesville, the thin-walled stainless tubes will produce turbine exhaust pressures equivalent to, or better than, the thicker-walled copper alloys at any given load and water velocities. In all respects, the differences in performance of stainless-steel tubes from that of copper alloys are definitely in favour of the stainless at Rivesville plant. Some of these advantages were entirely unexpected, it is true, but they exist neverthlesss. This, of course, does not mean that stainless-steel tubes are a universal answer. Where cooling-water conditions are such that satisfactory long life is expected from copper alloys, the evaluation of stainless steel may become much less attractive. However, where cooling water may result in severe corrosion of copper alloys, the possible use of stainless tubes must be considered.'

Corrosion Experience in Hydrogen-Fluoride Alkylation Units

D. J. BERGMAN and G. W. G. McDONALD: 'Solving Corrosion Problems in HF Alkylation Units.'

Corrosion, 1961, vol. 17, Apr., pp. 9-10, 12-13.

In developing its hydrogen-fluoride alkylation process from the initial laboratory stage and pilot-plant tests to its application in the full-scale commercial production of aviation gasoline component, the Universal Oil Products Company found that experience in handling anhydrous fluoride in process units was virtually non-existent, and that information on equipment and corrosion rates was available only from acid manufacturers and laboratory data. This review of the corrosion problems encountered, and the solutions evolved, is intended as a guide to operators of present-day alkylation units.

Reference is made to the initial laboratory and pilot-plant tests and to the metallurgical problems encountered in the operation of the first commercial-scale units. Carbon steel was early recognized as a suitable material of construction for the principal components of process units, but was not satisfactory in all applications. 'Monel', the use of which was ruled out initially, was later found satisfactory for use in trim and various kinds of valve.

Valves with steel cocks failed under attack by the hydrogen fluoride, but, eventually, use of 'Monel' and fluorocarbon plastics was found to eliminate packing and valve-seat problems. Steel bolts failed rapidly due to stress-corrosion cracking when small leaks resulted in acid attack. 'K Monel' bolts also failed, but 'Duranickel' bolts were found to give satisfactory service.

The dangers inherent in contamination of the process stream by water, or water by acid, are cited, and constant pH checks are advocated as a control measure.

Behaviour of Nickel-containing Steels and Alloys in Hot Ammonia Atmospheres

J. J. MORAN, J. R. MIHALISIN and E. N. SKINNER: 'Behaviour of Stainless Steels and Other Engineering Alloys in Hot Ammonia Atmospheres.'

Corrosion, 1961, vol. 17, Apr., pp. 191t-5t.

Although synthetic anhydrous ammonia is produced, sometimes handled, and, in certain applications, employed, at elevated temperatures, few quantitative data are available on the behaviour of metallic materials in hot ammonia atmospheres. In this paper the authors record the results of corrosion tests on specimens of 18 representative corrosion-resisting materials (1) exposed for 29,164 hours in the catalyst basket of an ammonia converter (operating at 490°-550°C. and a pressure of 5,200 p.s.i.) at a point where the ammonia concentration was 5-6 per cent., (2) placed (either in the pre-oxidized or polished condition) in a chemical-plant ammonia line and exposed to a gas temperature of 500°C. for 1,540 hours. After testing, specimens were subjected to metallographic examination.

The materials studied, and the corrosion data obtained from the tests in the ammonia converter, are reproduced in the table below. Over a number of years, other test racks were exposed in different ammonia-containing atmospheres, and to compare corrosion behaviour under varying nitriding conditions, corrosion data derived from nickel-containing specimens exposed in an ammonia heater and a nitriding furnace are plotted (together with those from the present study) as a function of the nickel content of the specimen.

Corrosion Behaviour in an Ammonia Converter

Material	Nominal Nickel Content %	Corrosion Rate (in./year)
Type 446 ..	—	0·00112
„ 430 ..	—	0·00090
„ 302B ..	10	0·00073
„ 304 ..	9	0·00059
„ 316 ..	13	0·00047
„ 321 ..	11	0·00047
„ 309 ..	14	0·00023
„ 310 ..	21	0·00014
37Ni/20Cr ..	37	0·00018
Type 314 ..	20	0·00010
„ 330 (*a*) ..	34	0·00006
„ 330 (*b*) ..	36	0·00002
80Ni/20Cr ..	80	—
60Ni/15Cr/Fe ..	60	—
'Inconel'	79	—
70Ni/20Cr/Fe ..	70	—
'Monel'	66	—
Nickel	99·5	—

The authors regard the results as evidence that the nickel-base alloys in the iron-nickel-chromium system are substantially more resistant to attack by nitrogen than those alloys richer in iron and chromium. It is suggested that nickel, when in combination with more strongly nitride-forming elements, acts primarily to reduce the solubility, and hence the diffusion rate, of nitrogen in the alloys, resulting in a pronounced reduction in the rate of corrosive attack. The results indicate that commercially pure nickel is not, on the other hand, necessarily immune to reaction with ammonia, and the extent of attack may be increased by other contaminants in the gas stream.

Selection of Materials for Marine Applications: Economic Considerations

F. L. LAQUE and A. H. TUTHILL: 'Economic Considerations in the Selection of Materials for Marine Applications.'

Paper Presented to the Soc. Naval Architects and Marine Engineers, New York Metropolitan Section, Feb. 23, 1961; 38 pp.

Ship owners are faced with replacement of much of the merchant fleet built during World War II, and economic considerations, rather than war-time exigencies, being now the controlling factor in the selection of materials of construction, cost and service experience, supplemented by test data, are once more of prime importance. Although records pertaining to the service life of materials of construction are seldom complete, those people responsible for repairs and overhauls can usually give a reasonable estimate of current experience. Data on service experience with piping, condensers, pumps and propellers have, through the co-operation of numerous ship owners, been obtained in this way and form the basis of discussion in the present paper.

The data, derived from records pertaining to 360 merchant ships and an even larger number of naval vessels, are summarized in terms of life in years, and the average life expectancy, of the various materials covered. These materials are listed below under the applications for which information is presented.

Piping

Service and Sanitary: Galvanized steel, red brass, copper, PVC, copper-nickel alloy, 70-30 and 90-10 copper-nickel-iron alloys.

Main Circulating: Galvanized steel, rubber-lined steel, copper, copper wiped with solder, aluminium-bronze, copper-nickel alloy.

Fire and Flushing: Steel, copper.

Deck Steam and Exhaust: Steel, copper.

Cargo: S.G. iron.

Cargo Heating Coils: Steel, aluminium-brass, S.G. iron.

Condensers

Tubes: Admiralty brass, aluminium-brass, 70-30 and 90-10 copper-nickel-iron alloys.

Tube Sheets: Muntz metal, Naval brass.

Water Boxes: Cast iron (variously coated), rubber-lined steel.

Propellers: Manganese-bronze.

Pumps Handling Sea Water: Case: bronze; Impeller: bronze; Impeller: 'Monel'.

The service data are compared with the results of sea-water corrosion tests on relevant materials, and this comparison, in conjunction with assessment of material costs and other significant factors, is used as a basis for estimating the economic advantages of using more durable materials in the applications covered.

The authors first discuss the factors involved in applying corrosion-test data to estimation of service life, and comment on data available on corrosion rates in sea-water and on resistance to pitting and to velocity and fouling effects. Economic aspects are then considered, and the general principles established are employed, in the following sections, to estimate the return on investment in piping, condenser, pump and propeller materials. Attention is drawn to the disparity between calculated and actual heat-transfer performance of cupro-nickel condenser tubes, and to the opportunity for a review of design factors that would take into account service performance.

On the basis of their review the authors draw the following conclusions:

'1. From service data on 360 commercial ships and naval experience, the average life expectancy may be reasonably estimated as:

Piping—Service and Sanitary	*Years*
Galvanized steel	5·7
Copper	5·9
PVC	possibly 20
90-10 Cu-Ni-Fe	20
70-30 Cu-Ni-Fe	20+

'2. From data on the rate of pitting in sea-water tests, the time to penetrate the minimum wall of sanitary piping may be estimated as:

Material	*Years*
Steel—Schedule 80	3–7
Copper—Schedule K	3¾–8
Red brass—Schedule 40	4¼–10¼

'3. A study of inside diameters indicates that cupro-nickel pipe (Mil T-16420) has virtually the same inside diameter as the next larger Schedule 80 pipe size from ½ in. to 3 in. I.P.S. Since steel and PVC are normally recommended and used in Schedule 80, to compare fairly cupro-nickel, steel and PVC piping, comparisons should be made at 'essentially equivalent inside diameters' rather than at equal nominal pipe sizes.

'4. 90-10 Cu-Ni-Fe piping is shown to be more economical in first cost and offers prospect of highest return on investment (31 per cent.) for service and sanitary piping when compared to PVC, 70-30 copper-nickel, copper and red brass on the basis of 'equivalent inside diameters'.

'5. From service data, the life expectancy of condenser tubes may reasonably be estimated as:

Material	*Years*
Aluminium brass	16 (46 per cent. retubed in 15 years or less)
90-10 Cu-Ni-Fe	Equal to 70-30 Cu-Ni-Fe
70-30 Cu-Ni-Fe (Commercial)	1865 (24 per cent. retubed in 15 years)
70-30 Cu-Ni-Fe (Naval)	20+

'6. Recently reported heat-transfer data on 70-30 Cu-Ni and aluminium-brass tubes in coastal power-plant condensers suggest that a 75 per cent. cleanliness factor for aluminium brass and a 90 per cent. cleanliness factor for cupro-nickel may be appropriate if substantial operating time in contaminated harbor waters is anticipated.

'7. Economic analysis indicates that a shipowner can expect a return on his investment when using 90-10 Cu-Ni-Fe or 70-30 Cu-Ni-Fe tubes.

'8. For those operators not faced with excessive costs from 'out-of-service' time, there is the very tempting opportunity to reduce initial costs of cupro-nickel condensers by using 18 gage tubes which are standard in land-based power plants.

'9. Service data suggest there is real economic justification for the use of alloys, such as nickel-aluminium bronze and austenitic stainless steel, more durable than manganese-bronze for propellers.'

Use of Cupro-Nickel Sheathing to Protect Power-Line Poles from Marine-Borer Attack

P. B. ROSS and D. B. ANDERSON: 'Cupro-Nickel Sheathing Prevents Marine-Borer Attack—Triple Pole Life Expected.'

Electric Light and Power, 1960, vol. 38, Oct. 1, pp. 68-70.

Confronted with the problem of preventing attack by marine boring organisms on power-line poles sited in marine locations, Carolina Power and Light Company, in conjunction with The International Nickel Company, Inc., initiated in September 1959 a test programme to determine the effectiveness of sheathing a preservative-treated pole with cupro-nickel sheet.

The minimum life of preservative-treated power poles in waters along the North Carolina coast is seven to ten years, during which period marine borers weaken the poles to such an extent that they cannot withstand the high winds that accompany severe storms. Every effort is made to ensure an uninterrupted supply of power to shore communities, and power poles have therefore to be frequently replaced. Numerous tests by The International Nickel Company, Inc., have demonstrated the exceptional resistance of cupro-nickel to corrosion by sea water, and an alloy containing copper 79, nickel 20, iron 1, per cent., was therefore selected as an ideal material for sheathing the test poles. The 12-mil-thick sheathing is expected to last at least 20 years before showing any evidence of salt-water corrosion, and may last as long as 40 years.

In this article economic factors are discussed, and details are given of the scope of the test programme, and the method used to sheath the pole and install it in its sea-water test site.

Lubricant for Use in Drawing Stainless Steels and Nickel-Chromium Alloys

'The Drawing of Stainless Steel and Nickel-Chrome Alloys.'

Wire Industry, 1961, vol. 28, Mar., pp. 261, 286.

In the drawing of stainless steels and nickel-chromium alloys, the work-hardening characteristics of the materials and the heat generated during drawing poses numerous problems, not the least of which is that of lubrication. Application of a lead coating, the method of lubrication most commonly used, is not altogether satisfactory (removal of the lead after drawing may involve acid pickling for a relatively long period, and, if this treatment is not completely effective, the lead residues can give rise to difficulties, for example, during bright annealing). With respect to the other metallic coatings usually employed for this purpose, reasonable adherence of copper and zinc to the basis metal is difficult to obtain, while cadmium is costly and the finish of the drawn product leaves much to be desired. In the light of this situation, Paroxite (London), Limited, have investigated the feasibility of developing a chemical coating which would equal the performance of metallic coatings and exhibit none of their disadvantages. This article summarizes the results of work carried out on an oxalate coating (which, designated 'BX', has been used successfully for the reduction of stainless-steel rod and wire prior to cold heading and spring forming) in an effort to provide a substitute for lead in multi-hole drawing.

This work culminated in the development of a lubricant deemed to offer the requisite characteristics (low moisture content, fine mesh, and stability at the operating temperatures). Testing under production conditions commenced in 1960, and development has reached the stage where 4 or 5 s.w.g. rod is currently being drawn to 10 or 11 s.w.g. at final speeds in the region of 450 ft./min. Reduction from 10 to 16 s.w.g. is achieved at 650 ft./min., and reduction from 16 to 22 s.w.g. wire at 850 ft./min. Intermediate sizes of rod are also being drawn.

The new lubricant is claimed to offer four advantages *vis-à-vis* lead or other metallic coatings: (1) costs are substantially reduced; (2) the technical disadvantages associated with the use of a lead coating are eliminated; (3) when the wire is subjected to ordinary annealing, the coating and lubricant are automatically dispersed, and, after cleaning, the wire has a better surface finish than with any other known coating (if the wire is to be bright annealed the coating can be removed by a 5-minute immersion in dilute acid); (4) the 'BX'-coated rod or wire can be drawn to exact shapes and tolerances.

Machining High-Strength Materials

L. A. HAUSER and M. C. METZGER: 'Problems of Machining Space-Age Metals.'

Metal Progress, 1961, vol. 79, Mar., pp. 97–100, 154, 156, 158, 160.

Fabrication of the ultra-high-strength steels heat-treated to Rockwell C50 and above poses considerable problems, while many of the high-temperature 'superalloys' are difficult to machine, irrespective of hardness. In this article the authors discuss the difficulties involved in machining such materials as low-alloy ultra-high-strength steels, precipitation-hardenable stainless steels, and nickel- and cobalt-base alloys, and refer to methods by which machining can be facilitated.

The following points are commented upon as of practical significance: the machine tools should be as rigid, up-to-date and 'overpowered' as possible; both the workpiece and the tool should be maintained rigid; tools should be kept sharp and the cutting edge should be honed slightly; carbide tools are often mandatory; in general, tools should be ground with a zero to slightly positive back rake (usually zero to 3°) and with a positive side rake (up to 6°); tool 'overhang' should be kept to a minimum to help in maintaining rigidity; use of heavy sulphur-base or sulphur-chlorinated lubricants is generally to be preferred; the depth of cut should be as heavy as possible to avoid 'glazing', and feeds and speeds should usually be lower than those used in machining conventional metals; drilling is the most difficult machining operation, and turning and facing, the easiest. The article contains brief notes on such unconventional methods as sub-zero machining, hot machining, chemical milling, spark-arc disintegration, and electrolytic machining, and includes a data sheet summarizing information on turning, milling, drilling and tapping the complex nickel-chromium precipitation-hardenable stainless steel 'A-286'.

Aluminium-containing Free-Machining Stainless Steel

'Aluminium in Stainless Alloy Improves Machinability.'

Iron Age, 1961, vol. 187, Mar. 16, pp. 118–9.

The sulphur content of A.I.S.I. Type 303 chromium-nickel stainless steel (0·15 per cent. minimum), while improving machining characteristics, tends to reduce corrosion-resistance, and sometimes causes splitting during fabrication or in service. This article gives details of a free-machining grade of stainless steel, designated 'Uniloy 303MA', which is claimed to combine excellent machinability with higher corrosion-resistance than Type 303 steel and no tendency to longitudinal splitting. Typical percentage compositions of the two steels are given in the table in the right-hand column.

Element	A.I.S.I. Type 303	'Uniloy* 303MA'
Carbon ..	0·1	0·1
Manganese ..	1·5	1·5
Silicon ..	0·5	0·5
Sulphur ..	0·25	0·13
Chromium ..	18·0	18·0
Nitrogen ..	9·0	9·25
Molybdenum	0·5	0·5
Aluminium ..	—	0·7

* U.S. Pat. 2,900,250

In 'Uniloy 303MA', which was developed by Universal-Cyclops Steel Corporation, the Type 303 composition is modified to give a lower sulphur content and to include 0·7 per cent. aluminium. These modifications reduce the deleterious effects of sulphur, increase tool life and give rise to smoother finishes. Machining rates are stated to be reduced by 15–40 per cent. As to the degree to which the problem of splitting has been overcome, cylindrical components of 'Uniloy 303MA' 0·16 in. (4 mm.) in internal diameter have been expanded to 0·25 in. (6·25 mm.) internal diameter without splitting, while similar parts in Type 303 steel split as they were being expanded to 0·185 in. (4·625 mm.) internal diameter.

Service experience in machining the steel is described in illustration of the technical and economic advantages it offers.

ANALYSIS

Determination of Nickel and Chromium in Mixtures without Prior Separation

See abstract on p. 143.

Spectrochemical Determination of Nickel in Waste Effluents from Plating Systems

See abstract on p. 147.

Voltammetric Determination of Cobalt and Nickel in Hard Magnetic Alloys

See abstract on p. 148.

Rapid Spectrophotometric Determination of Copper in Non-Ferrous Alloys

See abstract on p. 149.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Trade Marks*.

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